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**U.S. Army
Environmental
Center**

Decontamination Methods for Explosives-Contaminated Lagoon Rock

Report No. SFIM-AEC-TS-CR-94018
Contract No. DACA31-91-D-0079

DISTRIBUTION STATEMENT A
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February 1994

20070419391

Prepared for:
U.S. Army Environmental Center (USAEC)
SFIM-AEC-TSD
Aberdeen Proving Ground, MD 21010-5401

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94P-0814



REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank) Unclassified		2. REPORT DATE February 1994		3. REPORT TYPE AND DATES COVERED Final, 12/92 to 2/94
4. TITLE AND SUBTITLE Decontamination Methods for Explosives-Contaminated Lagoon Rock			5. FUNDING NUMBERS	
6. AUTHOR(S) Frye, Salacka, Martino, Lowe				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Roy F. Weston, Inc. 1 Weston Way West Chester, PA 19380			8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army Environmental Center SFIM-AEC-TSD Aberdeen Proving Ground, MD 21010-5401			10. SPONSORING / MONITORING AGENCY REPORT NUMBER SFIM-AEC-TS-CR-94018	
11. SUPPLEMENTARY NOTES				
DISTRIBUTION STATEMENT A				
12a. DISTRIBUTION / AVAILABILITY STATEMENT Unlimited			12b. DISTRIBUTION CODE	
<p style="text-align: center;">Approved for Public Release Distribution Unlimited</p>				
<p>13. ABSTRACT (Maximum 200 words)</p> <p>The U.S. Army Environmental Center has conducted a field demonstration of composting explosives-contaminated soils from Explosives Washout Lagoons at Umatilla Depot Activity (UMDA) in Hermiston, Oregon. The lagoons are lined with gravel and rocks which may have to be separated from soils prior to composting. Consequently, USAEC conducted this study to evaluate methods for decontaminating lagoon gravel and rocks. This testing was conducted in two phases. In the first test phase, aqueous washing using spray/screen and tumbler methods was evaluated. In the second phase, the potential enhancement achievable by the use of chemical washing solutions (aqueous solutions of surfactants or other reagents) was evaluated.</p> <p>The results of Phase I testing showed that aqueous washing alone could remove the majority of explosives contamination from lagoon gravel and rock. Hot washwater (140°F) may be more effective than lower temperature washwater (70°F) in removing explosives from the rock. The site specific cleanup criteria for UMDA (30 mg/kg TNT and 30 mg/kg RDX) were not achieved by water washing alone. However, testing in Phase II showed that a variety of chemical reagents, including biodegradable surfactants, could be used following initial water washing to achieve the lower required explosives levels on the rock and gravel. Initial explosives levels were lower in Phase II than in Phase I.</p> <p>In addition to these rock and gravel washing tests, a preliminary desk top evaluation of Hot Gas Decontamination for contaminated rock and gravel was conducted. This evaluation indicated that several existing equipment configurations may be adaptable to this use. Further testing of candidate equipment may be useful in confirming performance and developing design and operating criteria.</p>				
14. SUBJECT TERMS Rock, decontamination			15. NUMBER OF PAGES	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT	

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EXPLOSIVES - CONTAMINATED LAGOON ROCK**

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TABLE OF CONTENTS

<u>Section</u>	<u>Title</u>	<u>Page</u>
1	INTRODUCTION	1-1
1.1	Background	1-1
1.2	Objectives	1-2
2	AQUEOUS ROCK WASHING STUDY	2-1
2.1	Testing Program	2-1
2.1.1	Overview	2-1
2.1.2	Untreated Rock Characterization	2-3
2.1.2.1	General	2-3
2.1.2.2	Phase I	2-4
2.1.3	Screen Washing	2-4
2.1.3.1	Procedure	2-4
2.1.3.2	Sample Collection and Analysis	2-5
2.1.4	Tumbler Washing	2-6
2.1.4.1	Procedure	2-6
2.1.4.2	Sample Collection and Analysis	2-6
2.2	Test Results	2-7
2.2.1	Untreated Rock Characterization	2-7
2.2.1.1	Physical Characterization	2-7
2.2.1.2	Chemical Characterization	2-7
2.2.2	Screen Washing	2-9
2.2.3	Tumbler Washing	2-14
2.3	Conclusions	2-22
3	REAGENT ROCK WASHING STUDY	3-1
3.1	Testing Program	3-1
3.1.1	Overview	3-1
3.1.2	Untreated Rock Characterization	3-2
3.1.3	Water-Based Washing	3-2
3.1.4	Qualitative Screening Tests	3-4
3.1.4.1	Overview	3-4
3.1.4.2	Surfactants	3-5
3.1.4.3	pH-Adjusting Reagents	3-5
3.1.4.4	Other Chemical Reagents	3-8
3.1.4.5	Procedure	3-8
3.1.5	Reagent Washing	3-11
3.2	Test Results	3-11
3.2.1	Untreated Rock Characterization	3-11
3.2.1.1	Physical Characterization	3-11

TABLE OF CONTENTS
(Continued)

<u>Section</u>	<u>Title</u>	<u>Page</u>
	3.2.1.2 Chemical Characterization	3-13
	3.2.2 Water-Based Rock Washing	3-15
	3.2.3 Qualitative Screening Tests	3-24
	3.2.4 Reagent Washing	3-33
3.3	Conclusions	3-47
4	HOT GAS DECONTAMINATION EVALUATION	4-1
4.1	Introduction	4-1
4.2	Background	4-1
4.3	Identification of Potential Systems	4-2
4.4	Results	4-6
	4.4.1 Gas-Fired Shuttle Car Furnace	4-6
	4.4.2 Portable IRV Hydrocarbon Extractor	4-9
	4.4.3 Fluidized-Bed Heater	4-11
4.5	Conclusions	4-14
5	REFERENCES	5-1

LIST OF FIGURES

<u>Figure</u>	<u>Title</u>	<u>Page</u>
2-1	Particle Size Distribution Curve for Phase I Untreated Rock Sample	2-8
2-2	Screen Washing Tests: TNT Concentration in Rock Versus Wash Time	2-11
2-3	Screen Washing Tests: RDX Concentration in Rock Versus Wash Time	2-12
2-4	Screen Washing Tests: HMX Concentration in Rock Versus Wash Time	2-13
2-5	Tumbler Washing Tests: TNT Concentration in Rock Versus Number of Wash Cycles	2-17
2-6	Tumbler Washing Tests: RDX Concentration in Rock Versus Number of Wash Cycles	2-18
2-7	Tumbler Washing Tests: HMX Concentration in Rock Versus Number of Wash Cycles	2-19
3-1	Phase II Test Program Flow Chart and Sampling Program	3-3
3-2	Particle Size Distribution Curve for Phase II Untreated Rock Samples	3-14
3-3	TNT Concentration In Gravel Samples following Reagent Washing	3-36
3-4	RDX Concentration in Gravel Samples following Reagent Washing	3-37
3-5	HMX Concentration in Gravel Samples following Reagent Washing	3-39
4-1	Schematic Diagram for Process Equipment at HWAAP	4-3
4-2	Gas-Fired Shuttle Car Furnace Schematic	4-7
4-3	100 IRV Hydrocarbon Extractor System	4-10
4-4	Fluidized-Bed Heater Schematic	4-12

LIST OF TABLES

<u>Table</u>	<u>Title</u>	<u>Page</u>
2-1	Phase I Test Program Summary	2-2
2-2	Summary of Results for Screen Washing Tests	2-10
2-3	Summary of Results for Tumbler Washing Tests	2-15
3-1	Potential Surfactants for Reagent Washing	3-6
3-2	Qualitative Screening Test Method Summary	3-9
3-3	Reagent Washing Method Summary	3-12
3-4	Summary of Results for Water-Based Washing Tests	3-16
3-5	Washwater Characterization Results for TSS/TDS	3-17
3-6	Material Balance for Water-Based Washing Tests	3-19
3-7	TNT Mass Balance for Water-Based Washing Tests	3-21
3-8	RDX Mass Balance for Water-Based Washing Tests	3-22
3-9	HMX Mass Balance for Water-Based Washing Tests	3-23
3-10	Summary of Results for Qualitative Screening Tests	3-25
3-11	Summary of Results for Reagent Washing Tests: Gravel Samples	3-34
3-12	Summary of Results for Reagent Washing Tests: Liquid Samples	3-40
3-13a	Material Balance for Reagent Washing Tests: Cycle 7A	3-42
3-13b	Material Balance for Reagent Washing Tests: Cycle 7B	3-42

LIST OF TABLES
(Continued)

<u>Table</u>	<u>Title</u>	<u>Page</u>
3-14a	TNT Mass Balance for Reagent Washing Tests: Cycle 7A	3-44
3-14b	TNT Mass Balance for Reagent Washing Tests: Cycle 7B	3-44
3-15a	RDX Mass Balance for Reagent Washing Tests: Cycle 7A	3-45
3-15b	RDX Mass Balance for Reagent Washing Tests: Cycle 7b	3-45
3-16a	HMX Mass Balance for Reagent Washing Tests: Cycle 7A	3-46
3-16b	HMX Mass Balance for Reagent Washing Tests: Cycle 7B	3-46
4-1	Potential Vendors for Hot Gas Decontamination Systems	4-5

SECTION 1

INTRODUCTION

1.1 BACKGROUND

Roy F. Weston, Inc. (WESTON_®) conducted field studies for the U.S. Army Environmental Center (USAEC), formerly known as USATHAMA, to evaluate seeding and windrow composting for the treatment of explosives contaminated soils from the washout lagoons at the Umatilla Depot Activity (UMDA) in Umatilla, Oregon.

The explosives washout lagoons at UMDA are lined with gravel and rocks. If composting were to be used to remediate the lagoon soils, separation of the larger rocks may be necessary prior to treatment for safety reasons. In such a case, residual contamination on the larger rocks would have to be removed prior to their onsite redeposition.

Bench-scale studies using samples of the explosives washout lagoon rocks were conducted to evaluate the ability to wash residual explosives contamination from the rocks. Two phases of laboratory testing were conducted. In Phase I, two aqueous washing processes were evaluated as potentially viable technologies for the removal of explosives from the rocks: screen washing and tumbler washing. In Phase II, the potential enhancement achievable by the use of chemical washing solutions following initial aqueous washing was evaluated. The cleanup criteria specified for the UMDA lagoons (30 mg/kg TNT and 30 mg/kg RDX) were used as performance goals for the washed gravel fraction in these studies. No cleanup standard has been specified for HMX.

In addition to bench-scale testing of rock washing methods, a desktop evaluation of the potential use of hot gas decontamination methods to treat the lagoon rock was conducted.

1.2 OBJECTIVES

The primary objectives of the bench-scale rock washing treatability studies were to evaluate:

- The level of explosives contamination associated with the rocks following field screening to separate the rocks and soil.
- The potential effectiveness of selected washing operations in removing explosives contamination from the rocks.

The maximum concentration of TNT and RDX allowed in the rock after treatment is 30 mg/kg. A cleanup level has not been established for HMX.

The objectives of the desktop study of hot gas decontamination methods for the lagoon rock were to evaluate:

- The technical and economic feasibility of using the hot gas decontamination technique to decontaminate the residual explosives on the larger rocks.
- Whether field scale equipment and systems are currently available for use in the hot gas decontamination process for lagoon rocks.

The evaluation of hot gas decontamination for lagoon rock was based upon previous development by USAEC of hot gas decontamination for explosives contaminated equipment.

SECTION 2

AQUEOUS ROCK WASHING STUDY

2.1 TESTING PROGRAM

2.1.1 Overview

WESTON conducted a Phase I aqueous rock washing study utilizing two batch testing scenarios. The bench-scale testing program was conducted at WESTON's Environmental Technology Laboratory (ETL) located in Lionville, Pennsylvania. The various tasks of the study were performed during December 1992. Sample analyses were performed at WESTON's Analytical Laboratory in Lionville, Pennsylvania. The testing program for the rock washing treatability study is presented in Table 2-1.

The first washing scenario simulated a full-scale vibrating screen equipped with a pressurized water spray. The bench-scale program involved placing a measured quantity of rock on an oscillating screen, above a catch basin. The target water spray rate to rock volume was 20 gallons per minute per cubic yard (gpm/yd³). A pressurized water spray was evenly applied to the rock for a designated contact time. Three spray contact times (5, 10, and 15 minutes) and two spray temperatures (70°F and 140°F) were evaluated for a total of six test runs.

The second washing scenario simulated a submerged, agitated washing process, similar to a full-scale cement mixer. A known quantity of the contaminated rock was placed in a stainless steel vessel (tumbler) with a measured quantity of tap water immersing the rock. The target washwater to rock volume ratio was 100 gallons per cubic yard (gal/yd³). Washing of the rock was achieved by rotating the tumbler for a specified contact time. Following agitation, the rock was strained on a No. 200 sieve, returned to the tumbler, and the process repeated using fresh washwater. Contact times of 10 and 30 minutes were evaluated, each using six sequential washes.

Table 2-1

Phase I Test Program Summary

Wash Method & Test Number (1)	Water Wash Rate & Volume/Rock Ratio	Temperature (Degree F)	Contact Time (minutes)	Number of Analytical Samples (2)				
				Rock (3)		Gravel Only (4)	Sand Only (4)	Wash Water
				Initial	Final			
Screen 1	20 gpm/cubic yd	70	5	1	1	1*	1*	1*
Screen 2	20 gpm/cubic yd	70	10	1*	1*	1*	1*	1*
Screen 3	20 gpm/cubic yd	70	15	1	1	1	1	1
Screen 4	20 gpm/cubic yd	140	5	1	1	1*	1*	1*
Screen 5	20 gpm/cubic yd	140	10	1*	1*	1*	1*	1*
Screen 6	20 gpm/cubic yd	140	15	1	1	1	1	1
Tumbler 1	87 gal/cubic yd	70	10	1	1			1*
			10		1*			1*
			10		1	1	1*	1*
			10		1*			1*
			10		1*			1*
			10		1	1	1	1
Tumbler 2	90 gal/cubic yd	70	30	1	1			1*
			30		1*			1*
			30		1	1	1*	1*
			30		1*			1*
			30		1*			1*
			30		1	1	1	1
Duplicates and Blanks (5)					1			2
Total Number of Samples Collected				8	19	10	10	20
Total Number of Samples Analyzed				6	11	6	4	6

NOTES

- (1) Six screen washing tests were conducted at each wash rate, temperature and contact time specified. Each screen washing test was conducted on new (unwashed) untreated rock sample using fresh wash water. Two tumbler washing tests were conducted at each temperature and set of contact times specified. Each tumbler washing test included six consecutive washings of the same treatability rock sample using fresh water during each washing.
- (2) Rock, gravel only, sand only and wash water samples were analyzed for TNT, HMX, and RDX and total solids. Samples designated with a (*) suffix were only analyzed for total solids.
- (3) Rock samples were collected before washing and after washing for each specified contact time (i.e. two rock samples were collected for each screen test and seven rock samples were collected for each tumbler test).
- (4) Samples of the gravel only and sand only were also collected after washing for each screen test and after the final wash cycles 3 and 6 for each tumbler test.
- (5) Duplicate samples of the washed rock and wash water were collected for Screen Test 6. A blank sample of the wash water was also collected.

2.1.2 Untreated Rock Characterization

2.1.2.1 General

The untreated rock samples used for the Phase I and II rock washing studies represented a subset of the particle size fraction present in the rock lining the washout lagoons at UMDA. The rock represents reject material generated from the lagoon soil screening process used during the composting study. The rejected material contains primarily cobble and gravel size particles as defined below:

- Cobbles - diameters greater than 3"
- Gravel - diameters less than 3" and greater than 4.75 mm (No. 4 sieve)

The rejected material also contains a small fraction of sand and silt as described below:

- Sand - diameters less than 4.75 mm and greater than 0.075 mm (No. 200 sieve)
- Silt - diameters less than 0.075 mm

The untreated rock samples did not contain cobbles from the UMDA rock pile, but did contain gravel, sand and silt.

The distinction among these size fractions may be of significance in full-scale implementation of composting. It is likely that small material consisting of sand and silt can be returned to the compost along with washwater for treatment to achieve cleanup criteria. However, the cobble and gravel fraction may have to be excluded from the compost matrix due to safety and other operating requirements. Rock washing may contribute to the composting scenario both by separating contaminated sand and silt from the gravel and by removing explosives sorbed to or otherwise adhering to the cobble and gravel.

2.1.2.2 Phase I

For the Phase I rock washing study, approximately 75 gallons of rock sample was collected, using shovels, and placed in 15 five-gallon metal pails. Each pail was considered a

representative aliquot of the whole rock sample. In order to minimize differences in explosive concentration and particle size distribution between individual pails, the 15 pails were filled simultaneously in layers. One shovel full of rock from the rock pile was placed in each of the 15 pails before placing a subsequent shovel full in each pail. As the pails were filled, rocks with diameters greater than 3" were selectively omitted.

After collection, each pail was sealed, labeled, and shipped by common carrier according to DOT regulations. Upon arrival at the ETL, the untreated rock sample was logged-in and stored until testing. The "as received" bulk density and moisture content of the rock was measured on each of three pails selected randomly from the 15 pails received. Bulk density was determined by measuring the weight and volume of rock in each pail and calculating density (ASTM D2937). The moisture content was determined according to ASTM D2216. The particle size distribution of the rock was also measured on one of these three pails according to ASTM D421/422.

2.1.3 Screen Washing

2.1.3.1 Procedure

The screen washing test began by placing approximately 26 kilograms (16 liters) of rock sample on a 23" L x 15" W x 2.5" H, No. 4 vibratory sieve. The rock was washed using a 40 psi pressure spray at a wash rate of 1.6 liters per minute. A single spray nozzle was used to produce a 40-degree angle, sheet-spray pattern covering the width of the vibratory screen. The spray of washwater was manually swept across the length of the rock surface in even strokes at a height of 24 inches for the duration of the contact time. Contact times of 5, 10, and 15 minutes were evaluated each at washwater temperatures of 70°F and 140°F. Water for the 140°F tests was obtained from a heated water bath.

During each screen washing test, washwater along with the sand and silts, which passed through the No. 4 sieve were collected in a sloped trough. The trough contents drained into a No. 200 sieve. Following each test, washwater and silt, which passed through the sieve, were collected in a bucket, weighed, mixed, and sampled. The washed gravel retained on the No. 4 sieve and the washed sand retained on the No. 200 sieve were weighed and

sampled separately. The gravel and sand were then recombined, weighed, mixed, and sampled. A "fresh" aliquot of untreated rock material was used for each screen washing test run.

2.1.3.2 Sample Collection and Analysis

Prior to the start of each screen washing test, an initial sample of rock was collected to determine the concentration of explosives (TNT, HMX, and RDX) of the untreated material.

Samples were collected for explosives analysis and/or total solids content following each screen washing to evaluate the effectiveness of the treatment. Samples consisted of the rock (combined gravel, sand and silt), gravel only, sand only, and washwater (silt and water). This terminology will apply to all results presented in this report.

Table 2-1 presents a summary of the analytical testing performed for the screen washing evaluation. Explosive analyses were conducted using USATHAMA Method LW02 for the rock, gravel and sand samples and Method UW01 for the washwater samples. The use of USATHAMA Method LW02 for these samples required the following modifications.

- The use of a 10 gram sample as opposed to a 1 gram sample as specified in the method. This modification should provide a more representative sample as a 1 gram sample may be comprised of only one rock.
- The volume of extraction solvent (acetonitrile) was increased from 2 mL to 40 mL. This modification ensured that the entire sample was in contact with the extraction solvent.
- The extraction was completed by placing the extraction container (i.e., the bottle containing the sample and extraction solvent) into a cooled sonic bath for approximately 1 hour.

Only soluble TNT, RDX, and HMX were measured in the washwater. Method UW01 specifies that the washwater samples be filtered through a 0.45 micron filter and only the filtrate is analyzed for explosives.

2.1.4 Tumbler Washing

2.1.4.1 Procedure

The tumbler washing test began by placing approximately 14 kilograms (9 liters) of the rock sample in a 13" H x 11" D stainless steel open top tumbler angled at approximately 45 degrees. Four liters of tap water at 70°F were added to the tumbler to create a washwater to rock volume ratio of approximately 90 gal/yd³. An electrical motor turned the tumbler contents at approximately 50 rpm over the designated contact time. Two tests were conducted. One test evaluated six sequential wash cycles at 10 minute contact times. The other test evaluated six sequential cycles at 30 minute contact times.

For each test, at the end of each cycle, the contents of the tumbler were strained through a No. 4 and a No. 200 sieve stack. Water and solids passing the No. 4 and No. 200 sieve were collected into a bucket, weighed, mixed, and sampled. For Cycles 1, 2, 4 and 5, the sand retained on the No. 200 sieve and gravel retained on the No. 4 sieve were recombined, weighed and sampled before being returned to the tumbler. For Cycles 3 and 6, the washed sand and gravel fractions were weighed and sampled separately before recombining. After recombining the gravel and sand, four liters of fresh tap water were added, and the process was repeated until six wash cycles were completed.

2.1.4.2 Sample Collection and Analysis

Prior to the start of each tumbler washing test an initial sample of rock was collected to determine the concentration of explosives (TNT, HMX, and RDX) in the untreated material. A tap water sample was also collected and analyzed for explosives as a background check.

Samples were collected for explosives analysis and/or total solids content following each wash cycle to evaluate the effectiveness of the washing step. Samples consisted of the rock (combined gravel, sand and silt), gravel only, sand only, and washwater (silt and water). However, as noted in subsection 2.1.3.2, "washwater" data presented in this report represented the filtrate from the washwater samples. Table 2-1 presents a summary of the

analytical testing performed for the tumbler washing evaluation. Explosives analyses were conducted using the same methods used for the screen washing test samples.

2.2 TEST RESULTS

2.2.1 Untreated Rock Characterization

2.2.1.1 Physical Characterization

The untreated rock sample consisted of a grey and brown poorly graded gravel with 1.0 percent by weight (%) sand and 1.2% silt. The USCS designation of the material was GP. The gravel was made up grey and brown semi-rounded particles. The sand and silt fraction had a light red and brown color. The silt was observed to coat the surfaces of the gravel.

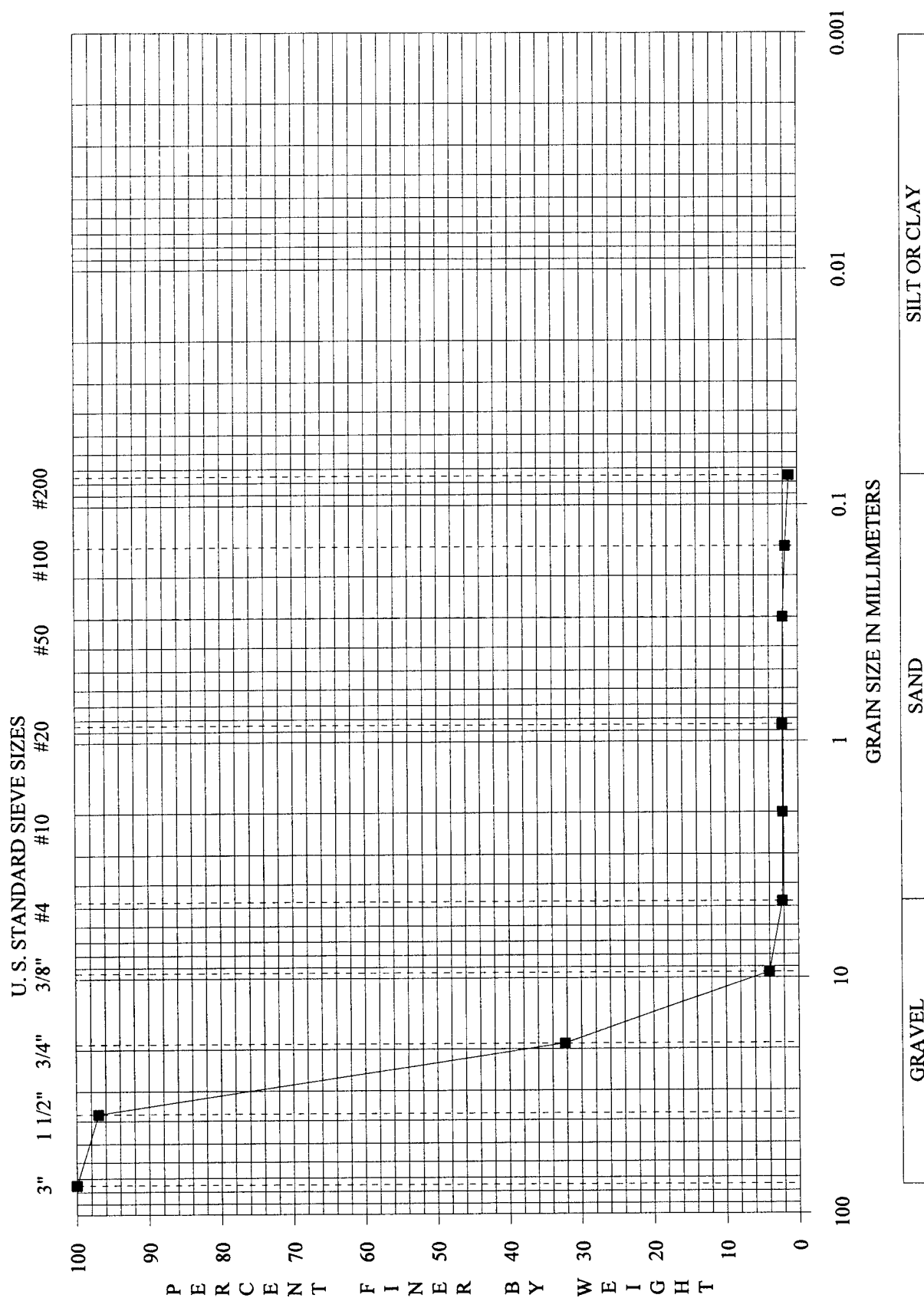
A particle size distribution curve for the rock samples is presented in Figure 2-1. Approximately 70% of the sample had particles larger than 3/4" diameter. The total percentage of sand and silt passing the No. 4 sieve (4.75 mm) was 2.2%.

The untreated rock sample had a total solids content of 98.7% and an "as received" bulk density of 99.2 pounds per cubic foot (pcf).

2.2.1.2 Chemical Characterization

The concentrations of explosives were measured in the untreated rock samples used for each of the various screen washing and tumbler washing tests. TNT concentrations in the untreated rock samples ranged from 1,610 mg/kg to 5,800 mg/kg with an average of 2,570 mg/kg. RDX concentrations in the untreated rock ranged from 417 mg/kg to 1,440 mg/kg, with an average of 657 mg/kg. HMX concentrations in the untreated rock ranged from 109 mg/kg to 393 mg/kg with an average of 175 mg/kg. In general, the relative proportions of explosives measured in the untreated rock were consistently 75% TNT, 20% RDX, and 5% HMX.

Figure 2-1 : Particle Size Distribution Curve for Phase I Rock Sample



2.2.2 Screen Washing

Results of the screen washing tests are presented in Table 2-2 and Figures 2-2 through 2-4. Results show that explosives concentrations in the rock were greatly reduced by screen washing. However, screen washing did not reduce TNT and RDX concentrations in the rock below the target maximum concentrations of 30 mg/kg. Specifically, final TNT concentrations ranged from 431 mg/kg to 1,210 mg/kg. Final RDX concentrations ranged from 105 mg/kg to 382 mg/kg. Final HMX concentrations ranged from 18 mg/kg to 65 mg/kg.

As discussed previously, the most important goal of the washing operation is to separate the sand and silt from the rock and to clean the large gravel fraction to meet the cleanup criteria. For the screen washing tests, TNT concentrations in the washed gravel ranged from 158 mg/kg to 259 mg/kg. RDX concentrations in the washed gravel achieved the target concentrations of 30 mg/kg and ranged from 16 mg/kg to 26 mg/kg. HMX concentrations ranged from 18 mg/kg to 20 mg/kg. Following washing, the silt coating observed on the untreated rock was removed yielding a gravel which appeared to be "clean" on the surface. The explosives concentrations observed in the washed gravel, therefore, indicate that these compounds are bound on or within the rock matrix.

TNT concentrations in the washed sand ranged from 15,500 mg/kg to 18,500 mg/kg. RDX concentrations in the washed sand ranged from 5,450 mg/kg to 7,720 mg/kg. HMX concentrations ranged from 826 mg/kg to 847 mg/kg. A comparison between explosives concentrations in the washed gravel and sand and the untreated rock, indicates that the explosives were effectively concentrated in the sand fraction. However, TNT and RDX concentrations in the gravel still exceeded the target maximum explosive concentrations.

A comparison between screen washed rock explosives concentrations and the washwater temperature (for either 5 and 15 minute wash times), presented in Figures 2-2 through 2-4, shows that approximately 50% lower explosive concentrations were achieved in the washed rock by using heated washwater at temperatures of 140°F. A comparison between washed rock explosive concentrations and wash time for each set of test runs conducted at the same

Table 2-2

Summary of Results for
Screen Washing Tests (1)

Parameters	Test Number			
	1	3	4	6
Wash Rate, gpm/yd ³	19.9	20.2	19.2	20.3
Wash Temperature, degree F	70	70	140	140
Wash Time, minutes	5	15	5	15
Rock and Sand				
TNT Concentration, mg/kg				
Untreated Rock	2,000	5,800	2,110	1,610
Washed Rock	824	1,210	431	434
Washed Gravel Only	NA	158	NA	259
Washed Sand Only	NA	15,500	NA	18,500
RDX Concentration, mg/kg				
Untreated Rock	571	1,440	573	417
Washed Rock	230	382	143	105
Washed Gravel Only	NA	15.5	NA	26.0
Washed Sand Only	NA	5,450	NA	7,720
HMX Concentration, mg/kg				
Untreated Rock	144	393	159	109
Washed Rock	40	65	28	18
Washed Gravel Only	NA	18.4	NA	20.1
Washed Sand Only	NA	826	NA	847
Washwater (2)				
TNT Concentration, mg/kg	NA	62.5	NA	103
RDX Concentration, mg/kg	NA	23.1	NA	40.0
HMX Concentration, mg/kg	NA	1.7	NA	2.2

(1) NA = not analyzed

(2) Concentrations reported on a soluble basis.

Figure 2-2
Screen Washing Tests: TNT Concentration in Rock versus Wash Time

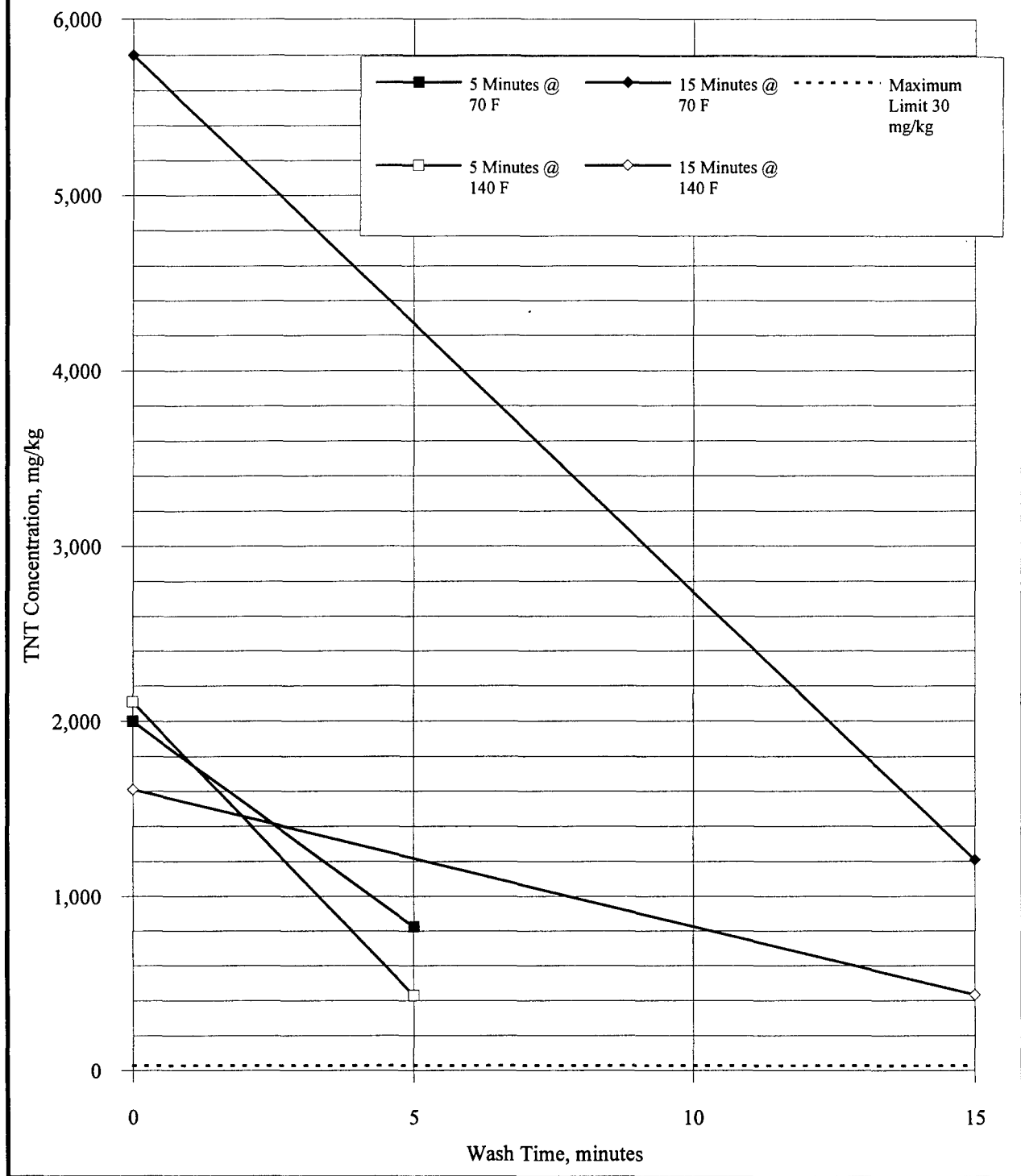


Figure 2-3
Screen Washing Tests: RDX Concentration in Rock versus Wash Time

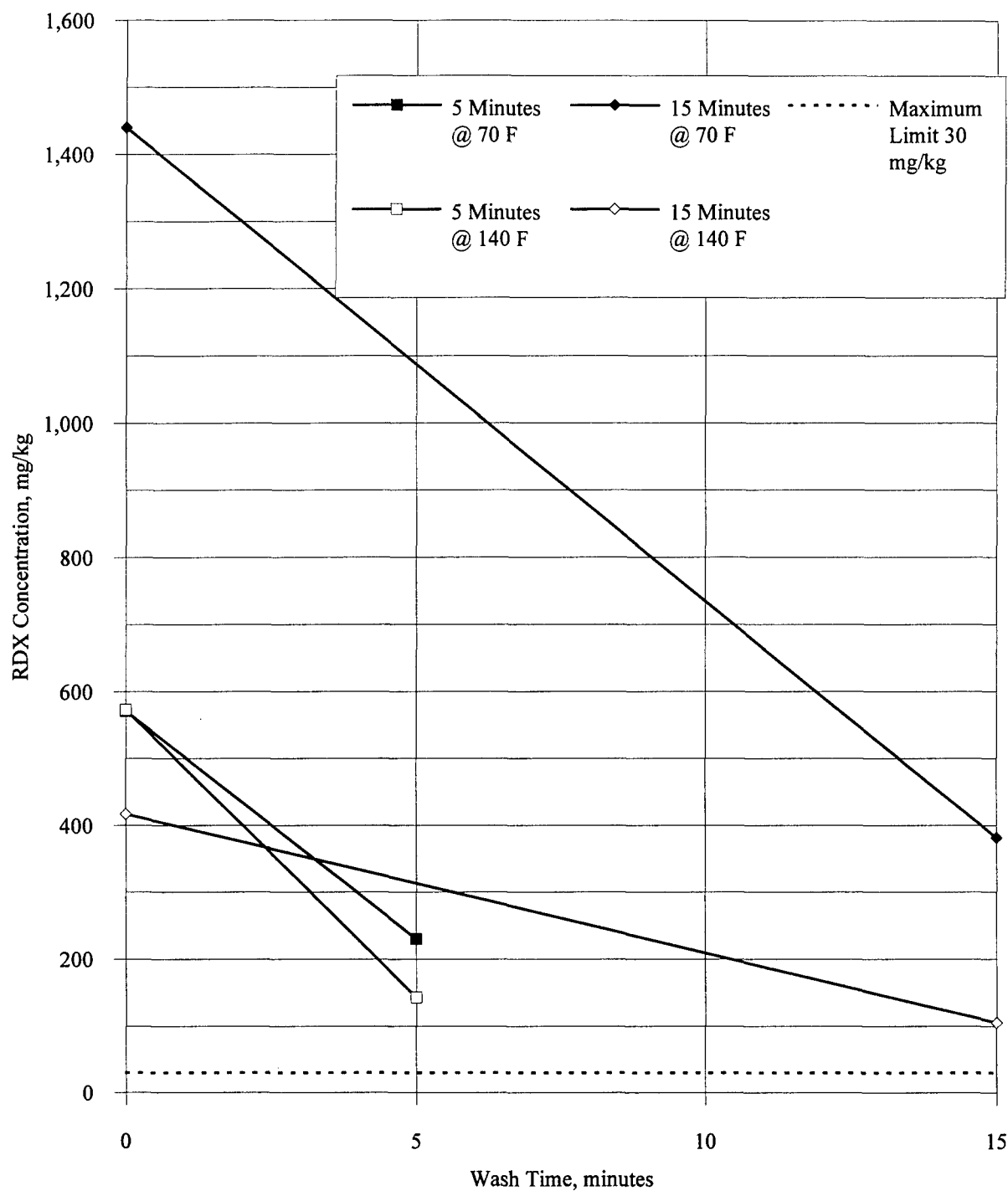
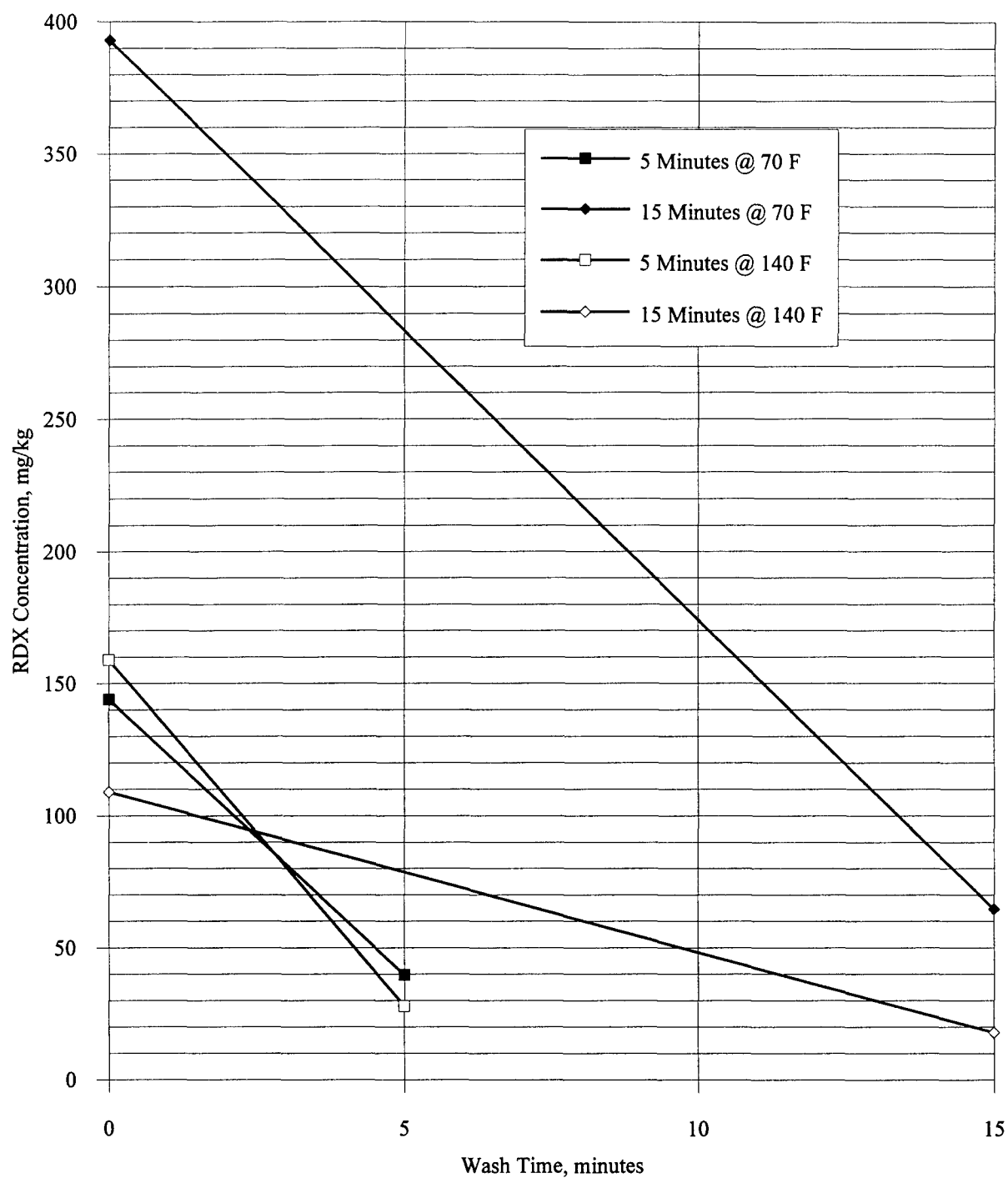


Figure 2-4
Screen Washing Tests: HMX Concentration in Rock versus Wash Time



washwater temperature shows that wash time had an insignificant effect on explosives removal using the screen wash method.

The explosives analysis on the washwaters are a measure of the soluble filtrate explosives concentrations. Soluble TNT concentrations, shown in Table 2-2, in the washwater ranged from 63 mg/kg in the 70°F washwater to 103 mg/kg in the 140°F washwater. Soluble RDX concentrations ranged from 23 mg/kg in the 70°F washwater to 40 mg/kg in the 140°F washwater. Soluble HMX concentrations ranged from 1.7 mg/kg in the 70°F washwater to 2.2 mg/kg in the 140°F washwater. Higher explosives concentrations were measured in the washwater from the 140°F tests, supporting the conclusion previously made, that heated washwater could be more effective at removing explosives from the rock.

2.2.3 Tumbler Washing

Results of the tumbler washing tests are presented in Table 2-3 and Figures 2-5 through 2-7. Tumbler washing test results show that explosives concentrations in the rock were greatly reduced at wash rates of 90 gal/yd³ after at least 3 cycles with contact times of 10 and 30 minutes. However, tumbler washing, even after six cycles, did not reduce the TNT concentrations below the target maximum concentration of 30 mg/kg. RDX concentrations in the Cycle 6 washed rock were below the maximum target concentration of 30 mg/kg.

Results of the tumbler washing test, using 10 minute wash cycles, show that the explosives concentrations in the mixed rock were reduced by approximately 50% after each wash cycle up to Cycle 3. TNT, RDX, and HMX concentrations in the Cycle 3 washed rock were 352 mg/kg, 83 mg/kg, and 20 mg/kg, respectively. After Cycle 3, no further significant reduction in TNT and HMX concentrations in the rock was observed (i.e., Cycle 6). However, the RDX concentration was reduced to 22 mg/kg (below the target concentration of 30 mg/kg).

As with the spray washing, the most important parameter for tumbler washing was the explosives concentrations in the washed gravel which would not be returned to the compost pile. For the tumbler washing test, using 10 minute wash cycles, TNT, RDX, and HMX

Table 2-3

Summary of Results for
Tumbler Washing Tests(1)

Parameters	Test Number	
	1	2
Wash Rate, gallons/yd ³ rock	86.8	89.7
Wash Temp., deg. F	70	70
Wash Cycle Time, minutes	10	30
Rock		
TNT Concentration, mg/kg		
Untreated Rock	2,250	1,670
Cycle 1 Washed Rock	760	829
Cycle 2 Washed Rock	868	559
Cycle 3 Washed Rock	352	206
Cycle 6 Washed Rock	321	119
Cycle 3 Washed Gravel	44.5	506
Cycle 6 Washed Gravel	322	115
Cycle 6 Washed Sand	1,300	798
RDX Concentration, mg/kg		
Untreated Rock	447	493
Cycle 1 Washed Rock	244	235
Cycle 2 Washed Rock	171	121
Cycle 3 Washed Rock	82.6	50.3
Cycle 6 Washed Rock	22.3	6.18 J
Cycle 3 Washed Gravel	12.9	33.0
Cycle 6 Washed Gravel	29.7	12.4 U
Cycle 6 Washed Sand	278	100
HMX Concentration, mg/kg		
Untreated Rock	118	127
Cycle 1 Washed Rock	53.4	51.4
Cycle 2 Washed Rock	47.6	22.4
Cycle 3 Washed Rock	20.1	8.4
Cycle 6 Washed Rock	25.0 U	15.1 U
Cycle 3 Washed Gravel	5.8	15.9
Cycle 6 Washed Gravel	13.6	16.1 U
Cycle 6 Washed Sand	21.0	7.8

Table 2-3
(continued)

Parameters	Test Number	
	1	2
Wash Rate, gallons/yd3 rock	86.8	89.7
Wash Temp., deg. F	70	70
Wash Cycle Time, minutes	10	30
Washwater (2)		
TNT Concentration, mg/kg		
Pre-Wash	0.00156 U	0.00156 U
Cycle 1	102	95.1
Cycle 2	100	88.0
Cycle 3	83.9	75.5
Cycle 6	58.6	65.6
RDX Concentration, mg/kg		
Pre-Wash	0.00126 U	0.00126 U
Cycle 1	27.0	34.9
Cycle 2	30.8	28.4
Cycle 3	26.8	26.8
Cycle 6	16.3	15.0
HMX Concentration, mg/kg		
Pre-Wash	0.00260 U	0.00260 U
Cycle 1	1.63	2.72
Cycle 2	2.21	2.77
Cycle 3	2.30	1.93
Cycle 6	2.12	2.73

(1) Data qualifier codes are defined below:

U = analyte was not detected at or below the reported practical quantification limit (PQL).

J = analyte was detected below the PQL, reported value is estimated.

NA = not analyzed

(2) Concentrations reported on a soluble basis.

Figure 2-5
Tumbler Washing Tests: TNT Concentration in Rock versus Number of Wash Cycles

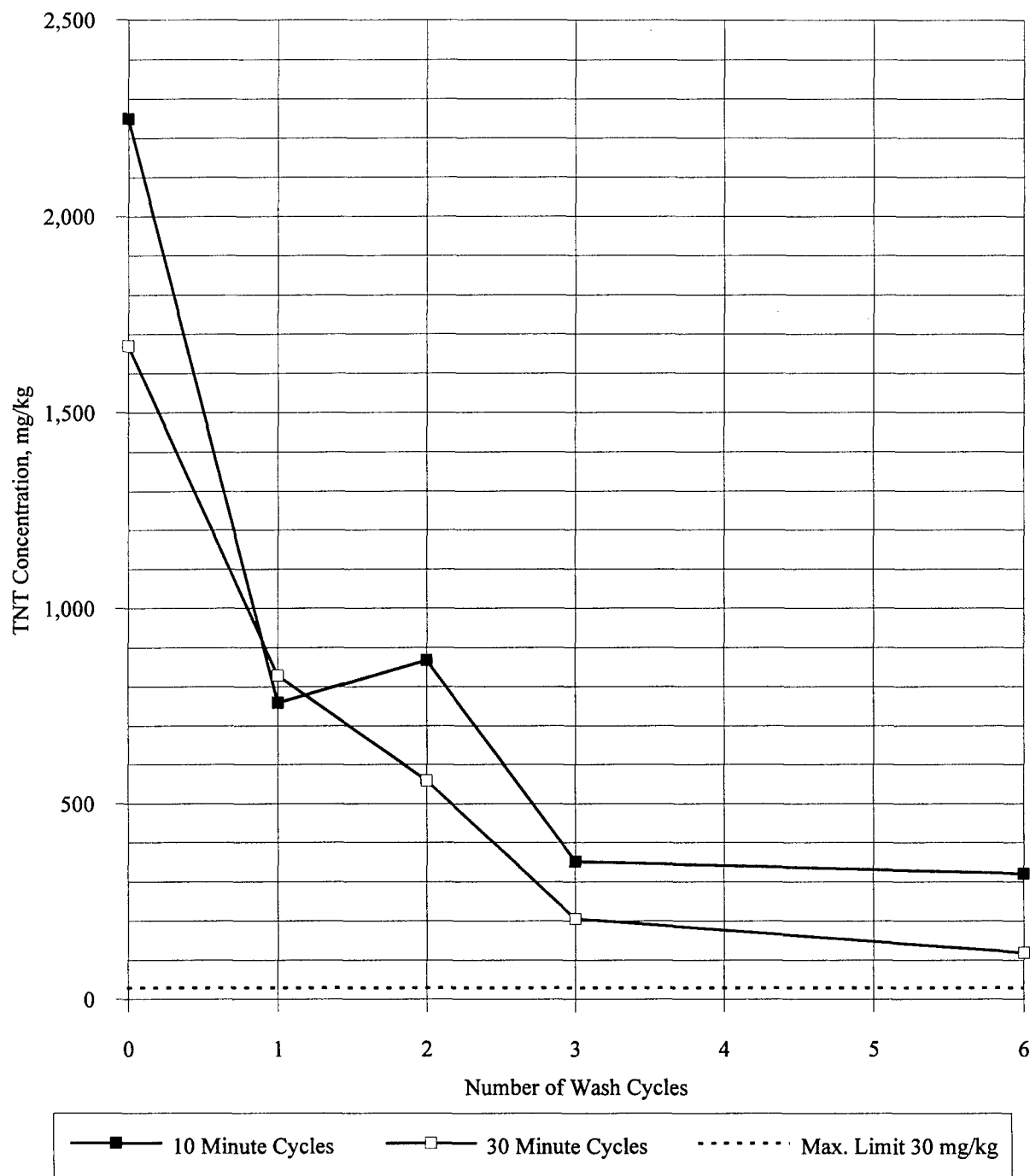


Figure 2-6
Tumbler Washing Tests: RDX Concentration in Rock versus Number of Wash Cycles

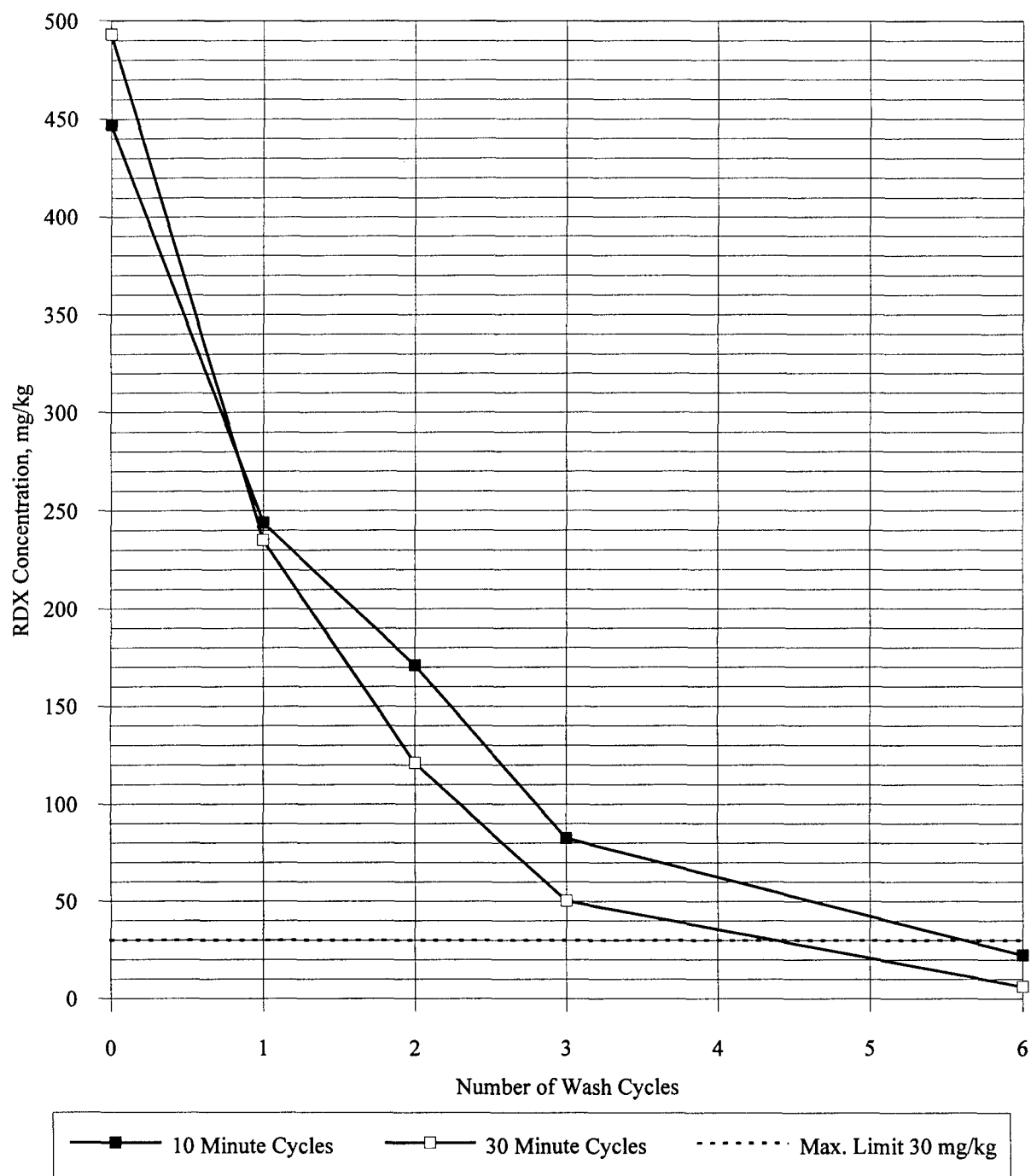
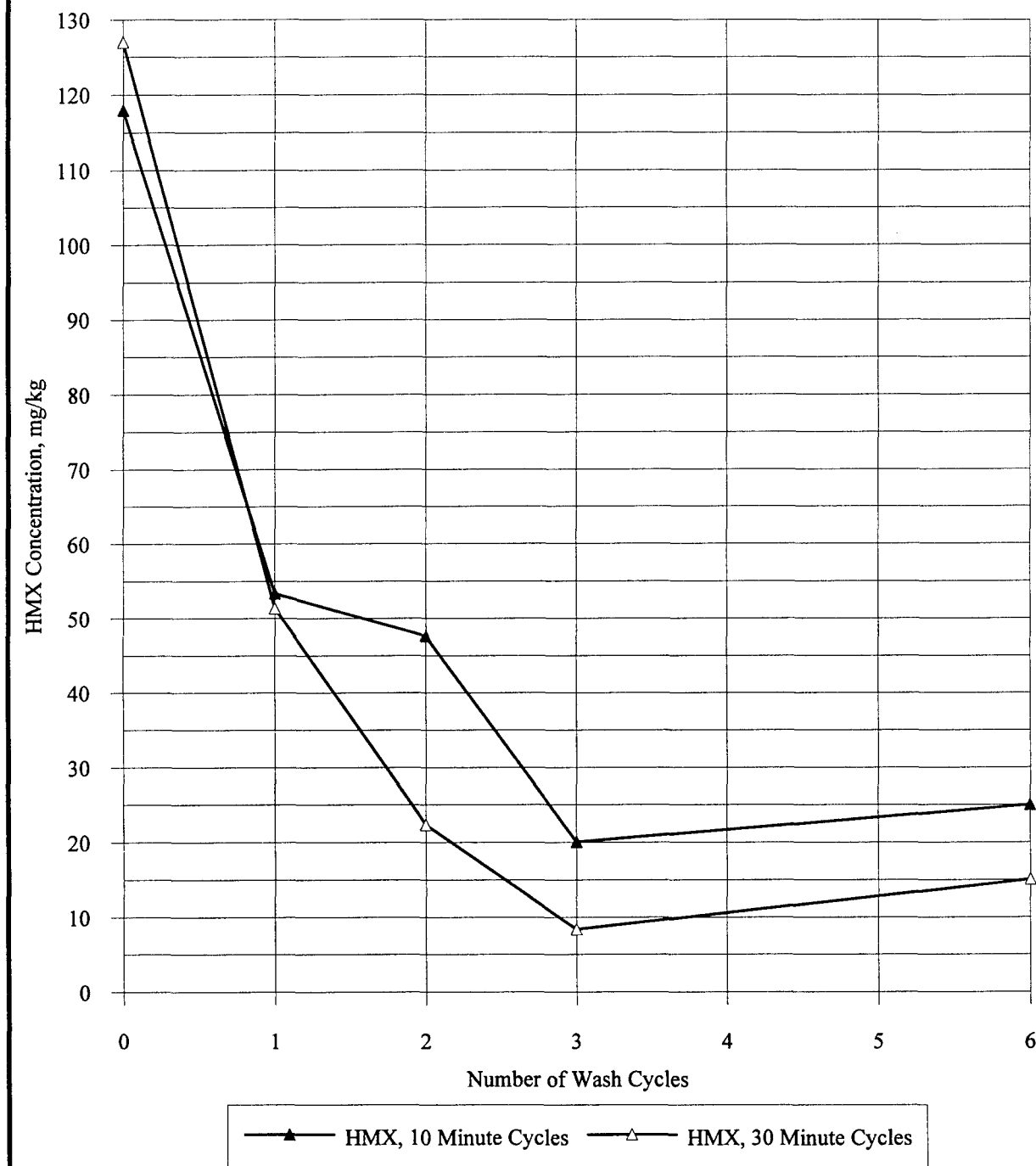


Figure 2-7
Tumbler Washing Tests: HMX Concentration in Rock versus Number of Wash Cycles



concentrations in the Cycle 3 washed gravel were 45 mg/kg, 13 mg/kg, and 5.8 mg/kg, respectively. TNT, RDX, and HMX concentrations in the Cycle 6 washed gravel were 322 mg/kg, 30 mg/kg, and 14 mg/kg, respectively. These data show that the maximum target concentration of 30 mg/kg was achieved for RDX but not TNT. TNT, RDX, and HMX concentrations in the Cycle 6 washed sand were 1,300 mg/kg, 278 mg/kg, and 21 mg/kg, respectively.

For the tumbler washing test, using 10 minute wash cycles, soluble TNT concentrations in the washwaters ranged from a high of 102 mg/kg after Cycle 1 to a low of 59 mg/kg after Cycle 6. Soluble RDX concentrations ranged from a high of 31 mg/kg after Cycle 2 to a low of 16 mg/kg after Cycle 6. Soluble HMX concentrations in the wash water ranged from a high of 2.3 mg/kg after Cycle 3 to a low of 1.6 mg/kg after Cycle 1.

Results of the tumbler washing test, using 30 minute wash cycles, showed that explosives concentrations in the rock were reduced by approximately 50% after each cycle up to Cycle 3. The concentrations of TNT, RDX, and HMX in the Cycle 3 washed rock were 206 mg/kg, 50 mg/kg, and 8.4 mg/kg, respectively. Between Cycles 3 and 6, explosive concentrations in the rock were reduced approximately 50%, indicating that less explosive was removed per cycle after Cycle 3. The concentrations of TNT, RDX, and HMX in the Cycle 6 washed rock were 119 mg/kg, 6.2 mg/kg, and less than 15 mg/kg, respectively. The target concentration for RDX was achieved in the Cycle 6 washed rock.

For the tumbler washing test, using 30 minute wash cycles, TNT, RDX, and HMX concentrations in the Cycle 3 washed gravel were 506 mg/kg, 33 mg/kg, and 16 mg/kg, respectively. TNT, RDX, and HMX concentrations in the Cycle 6 washed gravel were 115 mg/kg, <12 mg/kg, and <16 mg/kg, respectively. The target concentration for RDX was achieved for washed gravel from Cycles 3 and 6. TNT, RDX, and HMX concentrations in the Cycle 6 washed sand were 798 mg/kg, 100 mg/kg, and 7.8 mg/kg, respectively.

For the tumbler washing test, using 30 minute wash cycles, soluble TNT concentrations in the washwaters ranged from a high of 95 mg/kg after the Cycle 1 to a low of 66 mg/kg after Cycle 6. Soluble RDX concentrations ranged from a high of 35 mg/kg after Cycle 1 to a

low of 15 mg/kg after Cycle 6. Soluble HMX concentrations in the wash water ranged from a high of 2.8 mg/kg after Cycle 2 to a low of 1.9 mg/kg after Cycle 3.

A comparison between explosives concentrations in the washed gravel and sand indicate that (for both the 10 and 30 minute wash cycles) the explosives were concentrated in the sand fraction. In addition, the sand and silt, was observed to migrate to and concentrate at the bottom of the tumbler thus reducing the contact between the sand and/or silt particle surface and the washwater.

A comparison between tumbler washed rock explosives concentrations and wash cycle time shows that 30% to 60% lower explosive concentrations can be achieved in the washed rock and sand by using 30 minute wash cycle times. However, explosives concentrations in the washed gravel were generally higher for the 30 minute wash cycle than for the 10 minute wash cycle as compared on a cycle to cycle basis. These data suggest that although a 30 minute contact time was more effective in removing explosives from the rock and sand, a portion of the explosives remaining may have re-entrained back into the gravel. If significant quantities of sand are present in the full-scale tumbler washing system, the potential for re-entrainment of explosives back into the gravel may increase with increasing contact time.

2.3 CONCLUSIONS

The following conclusions summarize the results and key findings of the Phase I bench-scale aqueous washing treatability study for rocks from the explosives washout lagoons at UMDA:

Untreated Rock

- The untreated rock sample consisted of grey and brown poorly graded gravel with 1.0% sand and 1.2% silt. The untreated rock sample had a total solids content of 98.7% and an "as received" bulk density of 99.2 pcf.
- The average TNT, RDX, and HMX concentrations in the untreated rock sample were 2,570 mg/kg, 657 mg/kg, and 175 mg/kg, respectively. In general, the relative proportions of explosives measured in the untreated rock

sample were consistently 75% TNT, 20% RDX, and 5% HMX.

Screen Washing

- Screen washing at wash rates of 20 gpm/yd³, wash water temperatures of 70°F and 140°F, and contact times of 5 and 15 minutes, greatly reduced the explosives concentrations in the rock.
- Screen washing, did not reduce TNT concentrations in the washed rock or washed gravel below the target maximum concentrations of 30 mg/kg. The lowest final TNT concentration in the washed gravel was 158 mg/kg. This was accomplished in a wash temperature of 70°F at a 15 minute contact time. Screen washing reduced RDX concentrations in the washed gravel to concentrations below the 30 mg/kg target level.
- Screen washed gravel TNT concentrations ranged from 158 mg/kg to 259 mg/kg. RDX concentrations ranged from 16 mg/kg to 26 mg/kg. HMX concentrations ranged from 18 mg/kg to 20 mg/kg.
- For screen washing, soluble TNT concentrations in the washwater ranged from 63 mg/kg to 103 mg/kg. Soluble RDX concentrations ranged from 23 to 40 mg/kg. Soluble HMX concentrations in the washwater ranged from 1.7 mg/kg to 2.2 mg/kg. The lower concentrations of each explosive correspond to the 70°F washwater test and the higher concentrations correspond to the 140°F washwater test.
- Approximately 50% lower explosive concentrations in the washed rock were achieved by using heated wash water at temperatures of 140°F. Washtime did not have a significant effect on explosives removal.
- The surface of the washed gravel appeared to be visually "clean". The explosives compounds measured in the washed gravel, therefore, indicate that these compounds are bound within the rock matrix. The separated sand fraction contained higher concentrations of explosives the gravel fraction, indicating that separation based on particle size was responsible for much of the removal of explosives from the original rock.

Tumbler Washing

- Tumbler washing, at wash rates of 90 gal/yd³ after at least 3 cycles with contact times of 10 and 30 minutes, greatly reduced the explosives concentrations in the rock.
- Tumbler washing, did not reduce the rock or gravel TNT concentrations

below the target maximum concentration of 30 mg/kg. However, tumbler washing, reduced the rock RDX concentrations below the target maximum concentration of 30 mg/kg. These levels were achieved for RDX in the washed gravel after three cycles.

- For the tumbler washing test using 10 minute wash cycles, TNT, RDX, and HMX concentrations in the Cycle 3 washed gravel were 45 mg/kg, 13 mg/kg, and 5.8 mg/kg, respectively. TNT, RDX, and HMX concentrations in the Cycle 6 washed gravel were 322 mg/kg, 30 mg/kg, and 14 mg/kg, respectively. TNT, RDX, and HMX concentrations in the Cycle 6 washed sand were 1,300 mg/kg, 278 mg/kg, and 21 mg/kg, respectively. For the tumbler washing test using 30 minute wash cycles, TNT, RDX, and HMX concentrations in the Cycle 3 washed gravel were 506 mg/kg, 33 mg/kg, and 16 mg/kg, respectively. TNT, RDX, and HMX concentrations in the Cycle 6 washed gravel were 115 mg/kg, <12 mg/kg, and <16 mg/kg, respectively. TNT, RDX, and HMX concentrations in the Cycle 6 washed sand were 798 mg/kg, 100 mg/kg, and 7.8 mg/kg, respectively.
- For the tumbler washing test using 10 minute wash cycles, soluble TNT concentrations in the washwaters ranged from a high of 102 mg/kg after the Cycle 1 to a low of 59 mg/kg after Cycle 6. Soluble RDX concentrations ranged from a high of 31 mg/kg after Cycle 2 to a low of 16 mg/kg after Cycle 6. Soluble HMX concentrations in the washwater ranged from a high of 2.3 mg/kg after Cycle 3 to a low of 1.6 mg/kg after Cycle 1. For the tumbler washing test, using 30 minute wash cycles, soluble TNT concentrations in the washwaters ranged from a high of 95 mg/kg after the Cycle 1 to a low of 66 mg/kg after Cycle 6. Soluble RDX concentrations ranged from a high of 35 mg/kg after Cycle 1 to a low of 15 mg/kg after Cycle 6. Soluble HMX concentrations in the washwater ranged from a high of 2.8 mg/kg after Cycle 2 to a low of 1.9 mg/kg after Cycle 3.

SECTION 3

REAGENT ROCK WASHING STUDY

3.1 TESTING PROGRAM

3.1.1 Overview

Phase I aqueous washing tests demonstrated that aqueous washing alone would remove the majority of residual explosives on lagoon rock. Residual contamination after repeated washing cycles generally remained above the treatment criterion of 30 mg/kg TNT and 30 mg/kg RDX, although the RDX treatment criteria was achieved in some tests. It is possible that the residual explosives levels represent contaminants adsorbed to the rock (as opposed to those adsorbed to or entrained in the sand fractions), and that further removal may require dissolution/desorption of explosives into the washing solution.

As a result, Phase II testing was conducted to evaluate the enhancement in removal which might be obtained through the use of chemical reagents for washing. The Phase II study consisted of reagent rock washing using the tumbler method developed in Phase I. The selection of reagents to be tested was based upon the reagent's potential effectiveness for removal of explosives and its compatibility with biological processes (biodegradability or lack of toxicity) under the assumption that the washwater/solution would be returned to the compost pile to assist in moisture control and to provide treatment of the desorbed explosives.

The Phase II reagent rock washing study was conducted at the same WESTON laboratory facilities which performed the Phase I testing. Bench-scale treatability and physical testing were performed at WESTON's ETL and chemical analyses were performed at WESTON's Lionville Analytical Laboratory. The Phase II study was performed from July to September 1993.

The testing program for the Phase II study consisted of the following tasks:

- Untreated rock characterization.

- Water-based washing.
- Qualitative screening tests.
- Reagent washing.

Figure 3-1 presents a flow chart which summarizes the Phase II study. Each of the above tasks is described in the following subsections.

3.1.2 Untreated Rock Characterization

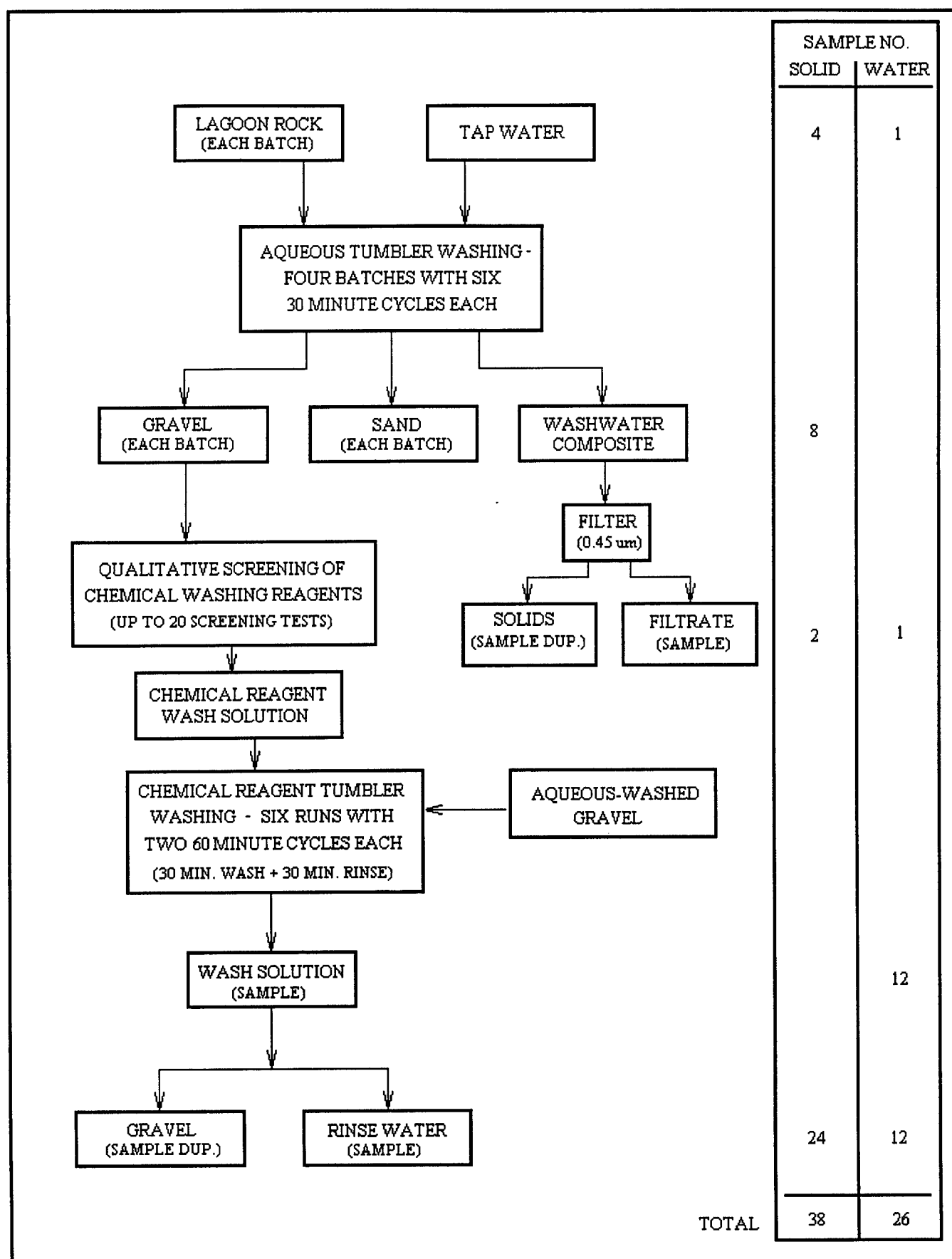
Twenty, 5-gallon containers of explosive-contaminated rock excavated from the UMDA washout lagoons were collected by WESTON personnel. The samples were shipped from UMDA by commercial carrier and were received at the ETL on 14 July 1993.

Approximately 70 kg of the as-received lagoon rock was mixed together and then divided into four representative fractions using a soil splitter. Two samples of the rock were collected for physical characterization. Each of these two rock samples was taken from a different fraction. Physical characterization consisted of bulk density, moisture content, and particle size distribution tests. Four samples (i.e., one from each fraction) were collected for chemical analysis. Chemical characterization consisted of explosives analysis (i.e, TNT, RDX, and HMX).

Physical characterization testing utilized the same methods employed in the Phase I study, as previously described in subsection 2.1.2.2. Chemical analysis for explosives during the Phase II testing was conducted using U.S. EPA Method 8330 rather than the USATHAMA Method LW02 previously used in Phase I. This change in analytical method was based upon comparative analysis of the extraction efficiency between the two methods conducted in support of the field demonstration study (1).

3.1.3 Water-Based Washing

The first step of the bench-scale testing program consisted of water-based washing of the rocks. Each of the four fractions of rock was washed on a batch basis using six, 30 minute



**FIGURE 3-1 : PHASE II TEST PROGRAM FLOW CHART
AND SAMPLING PROGRAM**

cycles. These washing cycles used the same apparatus and followed the same operating procedure employed for the Phase I tumbler washing study.

Following the 30 minute mixing period, the contents of the tumbler (rocks and washwater) were poured into a No. 4 and No. 200 sieve stack. The gravel remained in the No. 4 sieve and the sand remained in the No. 200 sieve. The washwater, which contained any solid material finer than the No. 200 sieve, passed through the sieve and was collected in a plastic container placed below the sieve. The wet gravel was returned to the tumbler, and fresh tap water added and the gravel was washed for 30 minutes. This procedure was repeated until a total of six wash cycles was completed for each batch of rocks.

Following the six wash cycles, samples of the gravel and sand from each of the four batches were collected for explosives analysis. The combined washwater from batches 1 through 4 was filtered to remove the fines (i.e., 0.45 micron filter). Duplicate samples of the fines and a single sample of the filtrate were collected for explosives analysis.

Following washing and sampling of each of the four rock batches, the washed gravel was air dried and composited. This single large composite batch of washed gravel was then split into four representative portions using the soil splitter. One of the four portions was then set aside to be utilized in the qualitative screening tests. The remainder of the rocks were then divided into six equal portions using the soil splitter. These six portions were used in the final reagent washing tests.

3.1.4 Qualitative Screening Tests

3.1.4.1 Overview

The second step of the Phase II study involved the identification and selection of chemical reagents with the capability of enhancing the effectiveness of water washing in this application. Based on the results of the Phase I study, it was anticipated that the majority of the residual explosives contamination on the rock would be removed via the water washing cycles. This was due to the essentially complete removal of the fine solids with which the explosive contamination was presumably associated. The use of chemical reagent-

based washing cycles following the aqueous-washing step was intended to desorb those explosives contaminants not effectively removed using water alone.

Three general types of chemical reagents were considered for use in this application:

- Surfactants
- pH adjusting reagents
- Other reagents, based on literature information

A discussion of the three categories of chemical reagents follows.

3.1.4.2 Surfactants

A variety of surfactant vendors were contacted to obtain information on potentially applicable surfactants. In addition, several literature reports were obtained on the use of surfactants for explosives wastewater treatment (2,3). The results of these efforts indicated that there is relatively little available information or experience on the use of surfactants for this application. As a result, several additional surfactants were added to the list in order to include in the test a relatively wide variety of chemical structures and ionic types. Table 3-1 provides a preliminary list of the recommended surfactants based upon vendor contact and literature review. The table also contains information obtained from vendors regarding the chemical nature of the surfactants, as well as biodegradability and cost data.

3.1.4.3 pH - Adjusting Reagents

Literature reports indicate that elevated pH may be of use in solubilizing explosives, although definitive data on the pH relationship has not been found. In addition there is evidence that elevated pH may result in partial decomposition of some explosives (4). While most of the identified decomposition products are expected to be innocuous in terms of composting, their presence should be considered. Sodium hydroxide and sulfuric acid were used to adjust solution pH to values ranging from 7 to 11 for evaluation in the qualitative screening study.

Table 3-1

Potential Surfactants for Reagent Washing

Product	Manufacturer	Type	Estimated Cost (\$/lb) (1)	Biodegradability
BIO SO FT S100	Stepan	Dodecyl Benzene Sulfonic Acid	0.99	Yes (2)
D-40	Stepan	Na-DDBSA	0.64	Yes (2)
WAC	Stepan	Na lauryl SO ₄	0.77	Yes (2)
SXS	Stepan	Na xylene sulfonate	0.49	Yes (2)
RENEX 30	ICI	Poe(12)Tri Decyl Ether	1.76	Yes (3)
TWEEN 20	ICI	Polysorbate 20	2.04	Yes (2)
SILICATE N	PQ	Na Silicate	0.25	NA (4)
MACOL OP-10	Mazer	POE (10) Octyl Phenol Ether	1.38	Yes (3)
AVANEL S 35	Mazer	Octyl Phenol - POE (3) Sulfonate	1.25	Yes (2)
AVANEL S 50	Mazer	C12-C15 - POE(15)- Sulfonate	1.36	Yes (2)
AVANEL S 30	Mazer	C12-C15 - POE(3)-Sulfonate	1.30	Yes (2)
AVANEL S 74	Mazer	C8-POE(3)- Sulfonate	1.31	Yes (2)
NEODOL 1-7	Shell	1° C11 Linear Ether(7) ALC	0.85	Yes (2)
NEODOL 91-6	Shell	1° C9-C11 Ether(6) ALC	0.85	Yes (2)
NEODOL 25-9	Shell	1° C12-C15 Ether(9) ALC	0.87	Yes (2)
DUOMEEN T	AKZO	Primary	1.17	Yes (2)
ARQUAD T-50	AKZO	Quaternary Amine	1.06	Yes (2)

Table 3-1
(continued)

Potential Surfactants for Reagent Washing

Product	Manufacturer	Type		Estimated Cost (\$/lb) (1)	Biodegradability
DUOQUAD T-50	AKZO	Quaternary Amine	Cationic	1.24	Yes (2)
LECITHAN	Alcolac	Phospholipid	Anionic	1.50	Yes (2)
MACOL TD 3	Mazer	Tri Decyl Alcohol POE (3) Ether	Nonionic	1.48	Yes (3)
MACOL TD 610	Mazer	Tri Decyl Alcohol POE (6) Ether	Nonionic	1.33	Yes (3)
MACOL TD 12	Mazer	Tri Decyl Alcohol POE (12) Ether	Nonionic	1.38	Yes (3)
DOWFAX 3B 2	DOW	Disulfonated Aromatic	Anionic	1.16	Yes (2)
DOWFAX 8390	DOW	Disulfonated Aromatic	Anionic	1.14	Yes (2)
MAKON 4	Stepan	POE Nonyl Phenol Ether	Nonionic	1.22	Yes (2)
MAKON 10	Stepan	POE Nonyl Phenol Ether	Nonionic	1.09	Yes (2)
MAKON 12	Stepan	POE Nonyl Phenol Ether	Nonionic	1.09	Yes (3)

- (1) Prices based on multi-drum quantities and do not include shipping costs.
(2) Documented in vendor literature.
(3) Based on telephone conversation with vendor.
(4) Not applicable.

3.1.4.4 Other Chemical Reagents

According to reference 4, TNT reacts with sellite (sodium sulfite) to form water soluble sulfonates, as shown below:



This chemical reaction has been used to purify TNT and is pH sensitive. Product yield rises sharply above pH 7.5 due to the formation of the water soluble complex of 2,4,6-TNT with sodium sulfite. Therefore, the use of sellite at elevated pH levels appeared to have promise for removing explosives from rock. Thus, treatment with sodium sulfite, both alone and in combination with alkaline pH adjustment, was evaluated.

Consideration was also given to the evaluation of organic solvents such as methanol. However, the use of solvents was ruled out by USAEC, consequently, these substances were not included in the bench-scale screening program.

3.1.4.5 Procedure

The procedure used for the qualitative screening tests is summarized in Table 3-2. In addition to the various chemical solutions evaluated, one control test, using tap water was included in the screening study.

The qualitative screening tests utilized approximately six times the water to rock ratio (i.e., 4 liters water per 15 kilograms rock) used in the previous tumbler washing tests. This six fold increase was used to approximate the total volume of water utilized in the six aqueous wash cycles, as well as to ensure an adequate water contact for the agitated rocks during the shaker test.

Table 3-2
Qualitative Screening Test Method Summary

- 1) Prepare Webster's reagent.
- 2) Prepare solutions of surfactants or other chemical treatments.
- 3) Collect a subsample of the aqueous washed rocks and sieve it to obtain the fraction larger than 4 mesh and smaller than $\frac{1}{4}$ ".
- 4) Place approximately 25 g rocks into 250 mL plastic bottles (i.e., opening of $\frac{1}{2}$ ").
- 5) Add 50 mL of prepared solution to bottle containing rocks.
- 6) Place bottles on shaker table and agitate overnight (approximately 18 hours).
- 7) Empty contents of bottle (rocks and solution) into 7 cm ceramic funnel placed over empty white plastic cup. Retain the solution in the cup (i.e., washate sample).
- 8) Rinse rocks in funnel with 50 mL of tap water. Hold funnel over another empty white plastic cup. Retain the tap water in the cup (i.e., rinsate sample).
- 9) Place rinsed rocks on paper towel to air dry approximately 30 minutes.
- 10) After rocks are dried, place on sheet of white paper for testing with Webster's reagent.
- 11) Test rocks with Webster's reagent. Wet cotton swab with solution and wipe rock surfaces - look for red color. Also place drops of solution on rocks to see if color bleeds onto white paper.
- 12) Divide washate into three 15 mL aliquots in white plastic cups. Add 2 mL Webster's reagent to one cup and observe color change. Save one aliquot for later use, if needed.
- 13) Divide rinsate into two 25 mL aliquots in white plastic cups. Add 2 mL Webster's reagent to one cup and observe color change.
- 14) Record observations.

All test solutions containing surfactants or sodium sulfite were made using tap water at reagent concentrations of 0.5% by weight. This concentration was selected to ensure that all surfactants were evaluated at solution concentrations above their respective critical micelle concentrations (CMC). The CMC is the concentration at which surfactant molecules aggregate into spherical structures known as micelles. The spherical micelle structure occurs in aqueous surfactant solutions such that the nonpolar (hydrophobic) portions of the individual surfactant molecules form the interior of the micelle, thus minimizing contact with water. The polar (hydrophilic) portions of the individual surfactant molecules that make up the micelle are oriented outward, thus maximizing contact with water. As a result, organic compounds that are immiscible with water can be removed from surfaces and dispersed in water. In order to achieve good detergent action in an aqueous system, the surfactant concentration should be at or above its CMC value. However, CMC values of surfactants vary widely depending on chemical structure, and can range from approximately 0.01% to greater than 0.5% by weight (5).

It is possible that in this application, the use of a 0.5% solution concentration of any given surfactant or other chemical reagent was not optimal (i.e., effective dosage lower than 0.5%). Wash reagent dosage optimization would likely result in more economical rock processing but was beyond the project scope.

The test protocol required the screening test to be conducted using two different colorimetric indicators (Webster's Reagent for TNT and diphenylamine/sulfuric acid for RDX). Webster's Reagent is known to be an effective colorimetric indicator for TNT-based explosives. Positive results are denoted by the development of a red color when the test reagent comes into contact with TNT. This reagent is employed by the Army to qualitatively determine whether equipment is contaminated with TNT. According to reference 4, diphenylamine/sulfuric acid provides a strong blue color when in the presence of RDX. Observed color changes on rocks and in aqueous samples were used to assign relative effectiveness rankings for each of the chemical reagents tested.

3.1.5 Reagent Washing

The results of the qualitative screening tests were used to identify the most effective reagents for further testing. The selected chemical reagents were used during the third and final step of the Phase II study. A total of five reagents and one tap water control were evaluated.

The procedure for the reagent washing tests is summarized in Table 3-3. Prewashed gravel was immersed in the reagent solution and tumble washed for 30 minutes. Following chemical washing, the gravel was tumbled for 30 minutes in a rinse step using tap water. The gravel was then air dried. This wash/rinse cycle was repeated once again so that for each chemical treatment, two complete wash/rinse cycles were performed. During each cycle, wash and rinse water samples were obtained and submitted for explosives and total suspended solids (TSS) analysis. Air-dried gravel samples were also collected at the end of each cycle and were submitted for explosives and total solids (TS) analysis.

3.2 TEST RESULTS

3.2.1 Untreated Rock Characterization

3.2.1.1 Physical Characterization

Physical characterization of the untreated rock sample was performed using duplicate samples. The rock samples consisted of a gray, poorly graded gravel with a USCS designation of GP. One sample indicated that the rock contained a sand fraction of 5% and a silt fraction of 1% while the other sample indicated a sand fraction of 3%.

Particle size distribution curves for the duplicate rock samples are presented in Figure 3-2. Approximately 65% to 70% of the sample had particles larger than $\frac{3}{4}$ " diameter. The total percentage of sand and silt passing the No. 4 sieve (4.75 mm) ranged between 3% and 6%.

The untreated rock sample had a total solids content of approximately 99.5% and on "as received" bulk density of approximately 105 pcf.

Table 3-3

Reagent Washing Method Summary

- 1) Prepare chemical wash solution by adjusting pH and/or dissolving chemical reagent in tap water as required.
- 2) Place batch of rocks in tumbler.
- 3) Add wash solution to tumbler in amount required to achieve ratio equivalent to 4 L wash solution to 15 kg rocks.
- 4) Tumble rocks in wash solution for 30 minutes.
- 5) Empty contents of tumbler into No. 4 mesh sieve placed above plastic container.
- 6) Weigh sieve and wet rock. Subtract weight of sieve to obtain wet rock weight.
- 7) Weigh wash solution and plastic container. Subtract weight of container to obtain weight of wash solution recovered.
- 8) Obtain samples of wash solution, send for TSS and explosives analysis.
- 9) Place wet rocks in tumbler.
- 10) Add tap water to tumbler in amount required to achieve ratio equivalent to 4 L liquid to 15 kg rocks. (i.e., add water at identical weight to that of wash solution weight added in step 3).
- 11) Repeat steps 4 to 8.
- 12) Place wet rocks in metal pan to air dry in hood.
- 13) Weigh pan and dried rocks. Subtract weight of pan to obtain dry rock weight.
- 14) Obtain duplicate samples of dried rocks, send for TS and explosives analysis.
- 15) Re-weigh pan and dried rocks. Subtract weight of pan to obtain dry rock weight.
- 16) Place dried rocks in tumbler.
- 17) Add wash solution to tumbler in amount required to achieve ratio of 4 L wash solution to 15 kg rocks. (This value is based on weight of dried rock after obtaining duplicate samples).
- 18) Repeat steps 5 to 15.

Grain size distribution plot showing Percent Finer versus Grain Size in Millimeters for two soil samples, WW10L1 and WW30L1. The plot includes U.S. Standard Sieve Sizes (3", 1 1/2", 3/4", 3/8", #4, #10, #20, #50, #100, #200) and Grain Size in Millimeters (100, 10, 1, 0.1, 0.01, 0.001). The legend indicates that the solid line with square markers represents WW10L1 and the dashed line with diamond markers represents WW30L1. The plot is divided into three regions: GRAVEL (above 75 mm), SAND (75 mm to 0.075 mm), and SILT OR CLAY (below 0.075 mm).

Grain Size (mm)	Sieve / Note	WW10L1 (%)	WW30L1 (%)
100	3"	100	100
75	1 1/2"	100	100
47.5	3/4"	70	65
37.5	3/8"	10	10
30	#4	5	5
20	#10	2	2
10	#20	1	1
7.5	#20	0.5	0.5
4.75	#40	0.2	0.2
2.5	#60	0.1	0.1
0.85	#200	0.05	0.05
0.425	#40	0.02	0.02
0.075	#200	0.01	0.01

Comparison of the physical characterization results for the Phase II study with those from the Phase I study indicate that the untreated rock samples used in the studies were very similar in terms of physical properties.

3.2.1.2 Chemical Characterization

Table 3-4 summarizes the chemical characterization results of the as-received, unwashed lagoon rocks and of a sample of unused washwater (i.e., tap water). The table also contains chemical characterization results of the washed rock solid samples collected after six cycles of water washing. The washed rock solid samples included gravel, sand, and fines. Results from the filtered composite washwater samples collected after six cycles of water washing are included in the table as well.

It can be seen that the TNT concentration in the unwashed (Cycle 0) lagoon rock samples from batches 1 to 4 varied considerably, and ranged from 82 to 500 mg/kg. The average TNT concentration of the unwashed lagoon rock was 252 mg/kg. RDX concentrations in unwashed lagoon rock samples ranged from 77 to 190 mg/kg, with an average RDX concentration of 120 mg/kg. HMX concentrations ranged from 35 to 81 mg/kg, with an average HMX concentration of 60 mg/kg. Based on the average levels of each explosive in the unwashed lagoon rock, the relative proportions of TNT, RDX, and HMX were determined to be 58%, 28%, and 14%, respectively. Table 3-4 also indicates that each of the analyzed samples was nearly moisture-free, with an average total solids value of 99.6%. The filtered washwater sample prior to washing (i.e., filtered tap water) contained no detectable concentration of TNT, RDX, or HMX, and had a TS value of 192 mg/L. This value represents the dissolved solids present in the water sample.

Comparison of the Phase II chemical analysis results of the as-received unwashed lagoon rock with the Phase I results shows that there were significant differences in the degree of explosives contamination of the rocks in the two batches. As reported in Subsection 2.2.1.2, average Phase I TNT levels were 2,570 mg/kg, or approximately ten times the amount of TNT measured in the batch of rocks used for the Phase II study. The average Phase I RDX concentration was 657 mg/kg, approximately five times the RDX concentration in the Phase

Table 3-4
Summary of Results for Water-Based Washing Tests

Test Component	Parameter (mg/kg)									
	Cycle 0					Cycle 6				
	TNT	RDX	HMX	TS (l)	TNT	RDX	HMX	TS (l)		
Unwashed Rock										
Lagoon Rock										
Batch 1	340	120	65	99.5	NA	NA	NA	NA	NA	NA
Batch 2	500	190	81	99.6	NA	NA	NA	NA	NA	NA
Batch 3	82	92	60	99.4	NA	NA	NA	NA	NA	NA
Batch 4	87	77	35	99.9	NA	NA	NA	NA	NA	NA
Average	252	120	60	99.6	---	---	---	---	---	---
Washed Rock										
Gravel										
Batch 1	NA	NA	NA	NA	52	17	3.8			99.2
Batch 2	NA	NA	NA	NA	76	20	16			99.9
Batch 3	NA	NA	NA	NA	60	18	17			99.0
Batch 4	NA	NA	NA	NA	130	25	13			98.6
Average	---	---	---	---	80	20	12			99.2
Sand										
Batch 1	NA	NA	NA	NA	240	140	26			99.7
Batch 2	NA	NA	NA	NA	320	140	21			97.3
Batch 3	NA	NA	NA	NA	300	170	27			99.5
Batch 4	NA	NA	NA	NA	350	200	29			99.7
Average	---	---	---	---	303	163	26			99.1
Fines										
Batch 1-4	NA	NA	NA	NA	1,500	3,800 B	1,800 B			96.6
Batch 1-4 Duplicate	NA	NA	NA	NA	1,700	4,500 B	2,100 B			97.9
Average	---	---	---	---	1,600	4,150 B	1,950 B			97.3
Washwater										
Batch 1-4	0.0013 U	0.00063 U	0.00063 U	192	43 B	25 B	6.6 B			207

"NA" indicates not analyzed.

"U" indicates parameter not detected.

"B" indicates parameter detected in blank.

(1) All TS values in % units except for washwater, which were expressed in mg/L.

II study rocks. Finally, the average HMX concentration of the rocks used in the Phase I study was 175 mg/kg, roughly three times the HMX concentration of the rocks used in the Phase II study. Thus, the rocks used in Phase I of the rock washing study exhibited much higher contamination levels than those used in Phase II.

3.2.2 Water-Based Washing

As previously noted, Table 3-4 summarizes the chemical characterization results for the washed rock following six cycles of water washing. Table 3-5 provides the results of additional analyses that were conducted to further characterize both the unfiltered and filtered composited washwater. The table contains triplicate TSS values ranging from 9,030 mg/L to 9,565 mg/L for the unfiltered washwater, as well as an average TSS value of 9,301 mg/L. A total dissolved solids analysis was performed on a sample of the washwater filtrate. This value of 205 mg/L is nearly identical to the TS value of 207 mg/L for the washwater filtrate (see Table 3-4).

Table 3-4 shows that after six 30 minute wash cycles using water, average gravel concentrations of TNT, RDX, and HMX all decreased compared to those of the unwashed rocks. TNT levels in the washed gravel samples from batches 1-4 ranged from 52 to 130 mg/kg, with an average of 80 mg/kg. This represents an average decrease of 68% relative to that of the average TNT value (252 mg/kg) in the unwashed rocks. However, the TNT concentration in the washed gravel still exceeded the established TNT cleanup criterion of 30 mg/kg. RDX levels in the washed gravel samples from batches 1-4 ranged from 17 to 25 mg/kg, with an average of 20 mg/kg. This represents an average decrease of 83% relative to that of the average RDX value (120 mg/kg) in the unwashed rocks. RDX levels in the individual gravel samples were all below the established RDX clean-up criterion of 30 mg/kg. HMX levels in the washed gravel samples from batches 1-4 ranged from 3.8 to 17 mg/kg, with an average of 12 mg/kg. This represents an average decrease of 80% compared to that of the average HMX value (60 mg/kg) in the unwashed rocks. No HMX cleanup criteria have been established.

Table 3-5
Washwater Characterization Results for TSS/TDS

Description	Parameter	Sample ID	Result
Unfiltered Washwater	TSS (mg/L)	Sample 1	9,565
		Sample 2	9,030
		Sample 3	9,310
		Average	9,302
Filtered Washwater	TDS (mg/L)	Sample 1	205

Table 3-4 shows that each of the samples from the washed sand fraction exhibited explosives levels that were higher than those of the corresponding washed gravel fraction samples. TNT concentrations in the washed sand samples from batches 1-4 ranged from 240 to 350 mg/kg, with an average of 303 mg/kg. RDX concentrations in the washed sand samples from batches 1-4 ranged from 140 to 200 mg/kg with an average of 163 mg/kg. HMX concentrations from batches 1-4 ranged from 21 to 29 mg/kg, with an average of 26 mg/kg.

According to Table 3-4, the composited fines fraction samples exhibited explosives levels that were greater than both the sand and gravel samples. TNT levels in the washed composited fines samples ranged from 1,500 to 1,700 mg/kg, with an average of 1,600 mg/kg. RDX levels in the washed fines samples ranged from 3,800 to 4,500 mg/kg, with an average of 4,150 mg/kg. HMX levels in the fines samples ranged from 1,800 to 2,100 mg/kg, with an average of 1,950 mg/kg.

These data indicate that the explosives compounds are more highly concentrated in the finer particle size fraction of the rock. These results suggest, as would be expected, that decreases in gravel explosives levels after water washing were due largely to separation of the smaller solid material (sand and fines) from the gravel, rather than from desorption or solubilization of explosives associated with the relatively large gravel fraction. The limited solubility of TNT in water (about 100 mg/L) supports this possibility. The composited washwater filtrate after six wash cycles contained 43 mg/L TNT, 25 mg/L RDX, and 6.6 mg/L HMX, and had a TS value of 207 mg/L.

Table 3-6 contains a material balance for each of the four batches of water washed rock, as well as an overall material balance based on the sums of all the quantities in washing process. The table lists both liquid (tap water) and solid (unwashed lagoon rock) input quantities for each rock batch. Output quantities for each stream (wash liquid, gravel, sand, and fines) are also listed separately for each rock batch. Recoveries are calculated for both liquid and solids fraction. Additionally, overall recoveries are presented.

It can be seen that the material balance for each of the individual rock batches, as well as that for the overall (total) rock batch was very good. Liquid recoveries ranged from 94.8%

Table 3-6
Material Balance for Water-Based Washing Tests

Parameter	Batch				
	1	2	3	4	Overall
Input (kg)					
Liquid	25.104	24.947	26.139	26.181	102.371
Gravel	15.518	15.747	16.015	16.246	63.526
Total	40.622	40.694	42.154	42.427	165.897
Output (kg)					
Liquid	24.050	24.732	24.766	24.823	98.372
Gravel	14.482	14.829	15.414	15.470	60.196
Sand	0.230	0.496	0.355	0.300	1.380
Fines	0.220	0.226	0.226	0.227	0.898
Solids Total	14.931	15.551	15.996	15.997	62.475
Overall Total	38.982	40.283	40.762	40.820	160.846
Recovery (%)					
Liquid	95.8	99.1	94.7	94.8	96.1
Solids	96.2	98.8	99.9	98.5	98.3
Overall	96.0	99.0	96.7	96.2	97.0

to 99.1%, while solid recoveries ranged from 96.0% to 99.0%.

Tables 3-7 to 3-9 summarize mass balances and % removal calculations for TNT, RDX, and HMX respectively for each of the water washed rock batches. Each table also contains an overall mass balance and % removal based on the sums of the quantities in the four individual balances

Table 3-7 lists the quantities of TNT (in mg) input via liquid and gravel. TNT output for each process stream is also listed. It is interesting to note that 61.2% of the TNT mass was associated with the solids output, while 38.8% was associated with the liquid output. It can also be seen that the TNT % recovery ranged from 34.4% to 250.9% for each individual rock batch, while the overall TNT recovery for the sums of quantities in the form individual balances was 68.7%. For each of the individual rock batches, overall TNT removal ranged from -42.3% to 85.7%, while that for the sums of the quantities in the form individual rock batches was 69.7%.

Table 3-8 summarizes the RDX mass balance for each of the four individual rock batches as well as for the sums of the quantities in the four batches. The table shows that 85.7% of the RDX mass was associated with the solids, and 14.3% was associated with the liquid. The RDX recovery ranged from 131.3% to 403.1% for the individual rock batches. The recovery for the sum of the quantities in the four rock batches was 227.1%. For each of the rock batches, overall RDX removal ranged from 69.1% to 90.1%, while that of the sums of the quantities in the four individual rock batches was 84.1%.

Table 3-9 summarizes the HMX mass balance for each of the four individual rock batches as well as for the sums of the quantities in the four batches. The table shows that 86.2% and 13.8% of the HMX mass was associated with the solids and liquid, respectively. The HMX recovery ranged from 90.3% to 220.8% for the individual rock batches. The recovery for the sum of the quantities in the form rock batches was 123.5%. Overall HMX removal ranged 64.6% to 94.5% for the individual rock batches, while the % HMX removal for the sums of the quantities in the four rock batches was 80.2%.

Table 3-7
TNT Mass Balance for Water-Based Washing Tests

Parameter	Batch				
	1	2	3	4	Overall
Input (mg)					
Liquid	0.0	0.0	0.0	0.0	0.0
Gravel	5276.1	7873.4	1313.3	1413.4	15876.1
Total	5276.1	7873.4	1313.3	1413.4	15876.1
Output (mg)					
Liquid	1034.2	1063.5	1064.9	1067.4	4230.0
Gravel	753.1	1127.0	924.9	2011.1	4816.1
Sand	55.1	158.6	106.6	105.0	425.3
Fines	351.4	361.3	361.8	362.7	1437.3
Solids Total	1159.5	1647.0	1393.3	2478.9	6678.6
Overall Total	2193.7	2710.5	2458.2	3546.3	10908.6
Overall Recovery (%)	41.6	34.4	187.2	250.9	68.7
Overall Removal (%)	85.7	85.7	29.6	-42.3	69.7

Table 3-8
RDX Mass Balance for Water-Based Washing Tests

Parameter	Batch				
	1	2	3	4	Overall
Input (mg)					
Liquid	0.0	0.0	0.0	0.0	0.0
Gravel	1862.2	2991.9	1473.4	1250.9	7578.4
Total	1862.2	2991.9	1473.4	1250.9	7578.4
Output (mg)					
Liquid	601.3	618.3	619.1	620.6	2459.3
Gravel	246.2	296.6	277.5	386.8	1207.0
Sand	2027.5	2076.1	2620.4	3094.1	9818.1
Fines	911.4	937.2	938.5	940.7	3727.9
Solids Total	3185.1	3309.9	3836.4	4421.5	14753.0
Overall Total	3786.4	3928.2	4455.6	5042.1	17212.3
Overall Recovery (%)	203.3	131.3	302.4	403.1	227.1
Overall Removal (%)	86.8	90.1	81.2	69.1	84.1

Table 3-9
HMX Mass Balance for Water-Based Washing Tests

Parameter	Batch				
	1	2	3	4	Overall
Input (mg)					
Liquid	0.0	0.0	0.0	0.0	0.0
Gravel	1008.7	1275.5	960.9	568.6	3813.7
Total	1008.7	1275.5	960.9	568.6	3813.7
Output (mg)					
Liquid	158.7	163.2	163.5	163.8	649.3
Gravel	55.0	237.3	262.0	201.1	755.5
Sand	376.5	311.4	416.2	448.6	1552.8
Fines	428.3	440.4	441.0	442.0	1751.7
Solids Total	859.8	989.1	1119.2	1091.8	4059.9
Overall Total	1018.6	1152.3	1282.7	1255.6	4709.1
Overall Recovery (%)	101.0	90.3	133.5	220.8	123.5
Overall Removal (%)	94.5	81.4	72.7	64.6	80.2

3.2.3 Qualitative Screening Tests

Table 3-10 presents a summary of the qualitative results from the chemical treatment screening study for the washate and rinsate samples. The colorimetric indicator results of the screening tests for the gravel samples are not included in the table since, in all cases, the rocks turned red upon application of Webster's reagent and there was no discernable difference between the chemical solutions evaluated. Thus, all of the screening test samples yielded positive test results in the presence of Webster's reagent and could not be used to assess the relative effectiveness of the chemical reagents that were included in the screening evaluation. All ratings in Table 3-10 are qualitative in nature and are based on the assumption that development of a red color in the washate and rinsate samples indicates desorption of TNT from rock surfaces and subsequent solubilization.

The following results and findings were obtained from this study based on qualitative observations:

- Webster's reagent was an effective indicator for the presence of TNT and gave positive results when used to test gravel washed using the control treatment (tap water) as well as each of the chemical treatments evaluated in the study. Therefore, no wash solution was completely effective in removing residual TNT from gravel surfaces. Because the sensitivity of Webster's reagent in the detection of TNT on solids is not definitively known, it is not known whether residual TNT levels are below the 30 ppm treatment criterion.
- Webster's reagent was effective in detecting the presence of explosives in aqueous (washate and rinsate) samples. Observation of these samples, both with and without Webster's reagent added, were used to determine the relative rankings of chemical treatments.
- Diphenylamine/sulfuric acid was not an effective indicator for the presence of RDX in this application since the characteristic blue color did not develop in any case tested including the contaminated, unwashed rocks.
- High pH (11) wash solutions appeared to be effective in solubilizing TNT. Lower pH (7 and 9) wash solutions were not as effective in solubilizing TNT.
- A sellite (sodium sulfite) /pH 9 treatment combination appeared to be effective in solubilizing TNT. Sellite without pH adjustment did not appear to be as effective in removing TNT from the gravel.

Table 3-10
Summary of Results for Qualitative Screening Tests

Medium	Test Condition	Control Test		Sellite Tests			
		Tap Water		Sodium sulfite		Sodium sulfite/pH 9	
		Appearance	Score	Appearance	Score	Appearance	Score
Washate	Without Webster's Reagent (2)	Brown	0	Brown	0	Brown with sl. red color	1
	With Webster's Reagent (1,3)	Sl. darker solids, supernatant clear	2	Sl. darker solids, supernatant clear	2	Sl. darker solids, supernatant clear	2
Rinsate	Without Webster's Reagent (4)	Slightly turbid	1	Slightly brown, turbid	1	Slightly hazy, sl. turbid	1
	With Webster's Reagent (1,3)	Sl. darker solids, supernatant clear	3	Darker solids, supernatant clear	3	Darker solids, supernatant clear	3
Total		--	6	--	6	--	7

(1) 2mL Webster's Reagent added to sample aliquot.

(2) Appearance of washate without Webster's Reagent rated as follows: 0 - brown; 1 - red.

(3) Appearance of washate and rinsate with Webster's Reagent as compared to samples without Webster's Reagent rated as follows: 1 - no/little difference; 2 - slightly darker; 3 - darker.

(4) Appearance of rinsate without Webster's Reagent rated as follows: 0 - clear, no/little turbidity; 1 - some turbidity; 2 - highest turbidity.

Table 3-10
(continued)

Medium	Test Condition	pH Tests					
		pH 7		pH 9		pH 11	
		Appearance	Score	Appearance	Score	Appearance	Score
Washate	Without Webster's Reagent (2)	Brown	0	Brown	0	Brown with sl. red color	1
	With Webster's Reagent (1,3)	Little difference, supernatant clear	1.5	Little difference, supernatant clear	1.5	Darker red solids, supernatant clear	3
Rinsate	Without Webster's Reagent (4)	V. slightly turbid	0.5	Clear	0	Slightly turbid	1
	With Webster's Reagent (1,3)	Sl. darker solids, supernatant clear	2	Sl. darker solids, supernatant clear	2	Darker solids, supernatant clear	3
Total		--	4	--	3.5	--	8

(1) 2mL Webster's Reagent added to sample aliquot.

(2) Appearance of washate without Webster's Reagent rated as follows: 0 - brown; 1 - red.

(3) Appearance of washate and rinsate with Webster's Reagent as compared to samples without Webster's Reagent rated as follows: 1 - no/little difference; 2 - slightly darker; 3 - darker.

(4) Appearance of rinsate without Webster's Reagent rated as follows: 0 - clear, no/little turbidity; 1 - some turbidity; 2 - highest turbidity.

Table 3-10
(continued)

Medium	Test Condition	pH Tests				Surfactant Tests	
		pH 11/Duoquad T-50		pH 11/Neodol 91-6		Avanel S150	
		Appearance	Score	Appearance	Score	Appearance	Score
Washate	Without Webster's Reagent (2)	Red-brown (turned color very quickly)	1	Red-brown	1	Brown	0
	With Webster's Reagent (1,3)	Sl. darker solids, supernatant clear	2	Darker solids, supernatant clear	3	Little difference, supernatant clear	1.5
	Without Webster's Reagent (4)	Slightly turbid	1	Slightly turbid	1	Clear	0
Rinsate	With Webster's Reagent (1,3)	Sl. darker solids, supernatant clear	2	Darker solids, supernatant clear	3	Supernatant clear	1
	Total	--	6	--	8	--	2.5

(1) 2mL Webster's Reagent added to sample aliquot.

(2) Appearance of washate without Webster's Reagent rated as follows: 0 - brown; 1 - red.

(3) Appearance of washate and rinsate with Webster's Reagent as compared to samples without Webster's Reagent rated as follows: 1 - no/little difference; 2 - slightly darker; 3 - darker.

(4) Appearance of rinsate without Webster's Reagent rated as follows: 0 - clear, no/little turbidity; 1 - some turbidity; 2 - highest turbidity.

Table 3-10
(continued)

Medium	Test Condition	Surfactant Tests					
		Biosoft D-40		Stepanol WAC		Dowfax 8390	
		Appearance	Score	Appearance	Score	Appearance	Score
Washate	Without Webster's Reagent (2)	Brown	0	Brown	0	Brown	0
	With Webster's Reagent (1,3)	Sl. darker solids, supernatant clear	2	Sl. darker solids, supernatant clear	2	Sl. darker solids, supernatant cloudy	2
Rinsate	Without Webster's Reagent (4)	Almost clear	0	Almost clear	0	Almost clear	0
	With Webster's Reagent (1,3)	Sl. darker solids, supernatant clear	2	Sl. darker solids, supernatant clear	2	Sl. darker solids, supernatant clear	2
Total		--	4	--	4	--	4

(1) 2mL Webster's Reagent added to sample aliquot.

(2) Appearance of washate without Webster's Reagent rated as follows: 0 - brown; 1 - red.

(3) Appearance of washate and rinsate with Webster's Reagent as compared to samples without Webster's Reagent rated as follows: 1 - no/little difference; 2 - slightly darker; 3 - darker.

(4) Appearance of rinsate without Webster's Reagent rated as follows: 0 - clear, no/little turbidity; 1 - some turbidity; 2 - highest turbidity.

Table 3-10
(continued)

Medium	Test Condition	Surfactant Tests					
		Silicate N		Renex 30		Makon 10	
		Appearance	Score	Appearance	Score	Appearance	Score
Washate	Without Webster's Reagent (2)	Light brown	0	Brown	0	Brown	0
	With Webster's Reagent (1,3)	Little difference, supernatant clear	1.5	Sl. darker solids, supernatant clear	2.5	Sl. darker solids, supernatant clear	2.5
	Without Webster's Reagent (4)	Clear	0	Almost clear	0	Almost clear	0
Rinsate	With Webster's Reagent (1,3)	Little difference, supernatant clear	1.5	Sl. darker solids, supernatant clear	2	Sl. darker solids, supernatant clear	2
	Total	--	3	--	4.5	--	4.5

- (1) 2mL Webster's Reagent added to sample aliquot.
 (2) Appearance of washate without Webster's Reagent rated as follows: 0 - brown; 1 - red.
 (3) Appearance of washate and rinsate with Webster's Reagent as compared to samples without Webster's Reagent rated as follows: 1 - no/little difference; 2 - slightly darker; 3 - darker.
 (4) Appearance of rinsate without Webster's Reagent rated as follows: 0 - clear, no/little turbidity; 1 - some turbidity; 2 - highest turbidity.

Table 3-10
(continued)

Medium	Test Condition	Surfactant Tests					
		Macol TD 610		Tween 20		Neodol 91-6	
		Appearance	Score	Appearance	Score	Appearance	Score
Washate	Without Webster's Reagent (2)	Brown with sl. red color	1	Brown with sl. red color	1	Brown	0
	With Webster's Reagent (1,3)	Sl. darker solids, sl. red color, cloudy supernatant	2.5	Sl. darker solids, sl. red color, clear supernatant	2.5	Sl. darker solids, supernatant clear	2
Rinsate	Without Webster's Reagent (4)	Sl. turbid,	1	Sl. turbid,	1	Clear	0
	With Webster's Reagent (1,3)	Sl. darker solids, supernatant clear	2	Darker solids, supernatant clear	3	Sl. darker solids, supernatant clear	2
Total		--	6.5	--	7.5	--	4

(1) 2mL Webster's Reagent added to sample aliquot.

(2) Appearance of washate without Webster's Reagent rated as follows: 0 - brown; 1 - red.

(3) Appearance of washate and rinsate with Webster's Reagent as compared to samples without Webster's Reagent rated as follows: 1 - no/little difference; 2 - slightly darker; 3 - darker.

(4) Appearance of rinsate without Webster's Reagent rated as follows: 0 - clear, no/little turbidity; 1 - some turbidity; 2 - highest turbidity.

Table 3-10
(continued)

Medium	Test Condition	Surfactant Tests			
		Duoquad T-50		Arquad T-50	
		Appearance	Score	Appearance	Score
Washate	Without Webster's Reagent (2)	Red-brown	1	Red-brown	1
	With Webster's Reagent (1,3)	Little difference, supernatant clear	1.5	Sl. darker solids, sl. red color, clear supernatant	3
Rinsate	Without Webster's Reagent (4)	Sl. turbid, brown, looks dark	1.5	Sl. turbid, brown, looks dark	1.5
	With Webster's Reagent (1,3)	Sl. darker solids, supernatant clear	2	Darker solids, supernatant clear	2.5
	Total	--	6	--	8

(1) 2mL Webster's Reagent added to sample aliquot.

(2) Appearance of washate without Webster's Reagent rated as follows: 0 - brown; 1 - red.

(3) Appearance of washate and rinsate with Webster's Reagent as compared to samples without Webster's Reagent rated as follows: 1 - no/little difference; 2 - slightly darker; 3 - darker.

(4) Appearance of rinsate without Webster's Reagent rated as follows: 0 - clear, no/little turbidity; 1 - some turbidity; 2 - highest turbidity.

- Several surfactants appeared to be effective in solubilizing TNT. The most effective treatments were surfactant/pH 11 combinations. When used alone, the most effective surfactants were cationic in nature (average cationic surfactant rating:7). Nonionic surfactants appeared to be less effective (average nonionic surfactant rating: 5.4), while anionic surfactants were judged to be least effective (average anionic surfactant rating: 3.5).
- All surfactant and surfactant/pH adjustment treatment combinations included in the study displayed a tendency to foam upon vigorous shaking. None of the non-surface active materials tested (sodium sulfite, sodium silicate N, pH adjustment using caustic or sulfuric acid) exhibited any foaming tendencies.
- The Duoquad T-50/pH 11 treatment sample began to darken and display a red color within minutes of contacting the wash liquid with the gravel. This was the most dramatic and fastest color change noted.
- With few exceptions, addition of Webster's reagent to washate and rinsate samples typically resulted in flocculation and settling of fine suspended solids, and very clear supernatants.

Based on the results of the qualitative screening test, WESTON recommended that the following chemical treatments be included in the final series of rock washing tests utilizing various chemical reagents:

- Control (Tap water).
- pH 11 (Of the three different pH levels evaluated, chemical treatment consisting of pH adjustment to 11 was rated the highest).
- 0.5% sellite/pH 9 (Sellite was screened both alone and in combination with pH adjustment to 9. The sample which was adjusted to pH 9 was rated higher in the screening study. Additionally, Reference 2 indicates that TNT may be solubilized by sellite at pH levels greater than 7.5.)
- 0.5% Arquad T-50 (One of the highest rated surfactants screened).
- 0.5% Neodol 91-6 / pH 11 (One of the highest rated surfactant-based treatments screened).
- 0.5% Duoquad T-50 / pH 11 (Other surfactant treatments were rated higher; however, this treatment was observed to cause the wash solution to exhibit a reddish tint within minutes, thus indicating that it may be effective in solubilizing TNT).

Based on USAEC knowledge and experience, Arquad T-50 and Duoquad T-50 have exhibited toxicity problems in the past. Tween 20 and Macol TD 610 were selected by USAEC personnel to replace Arquad T-50 and Duoquad T-50 due to their high ranking in the qualitative screening test results.

3.2.4 Reagent Washing

Table 3-11 contains the results of chemically washed gravel samples that were characterized. For each of the six chemical reagent evaluation tests that were conducted, two duplicate gravel samples per wash cycle were collected and analyzed. TNT, RDX, and HMX concentrations, as well as TS levels, were determined. The table shows that after one wash cycle the control (tap water washed) sample exhibited an average TNT level of 67 mg/kg. This value was only slightly lower than the average TNT level of the gravel after washing with six sequential water cycles (80 mg/kg). The two individual TNT sample values used to calculate the average of 67 mg/kg were 120 mg/kg and 14 mg/kg. This represents a very wide variation between these two sample values. It is not known which (if either) value is inaccurate.

Table 3-11 also shows that gravel samples treated with each of the chemical washing reagents exhibited average TNT levels ranging from 7.3 mg/kg to 27 mg/kg after one chemical wash cycle. These values are below the TNT cleanup goal of 30 mg/kg. It should be noted that the Cycle 7A duplicate TNT test values for the pH 11 and Macol TD 610 treatments were 36 and 44 mg/kg, respectively, and that each of the remaining individual TNT test values were within the 30 mg/kg TNT limit.

The average Cycle 7B TNT concentrations for gravel treated using the tap water control as well as each of the chemical reagents ranged from 11 mg/kg to 21 mg/kg and were below the 30 mg/kg TNT limit. With one exception (Macol TD 610, individual Cycle 7B TNT concentration was 31 mg/kg), all of the individual Cycle 7B TNT results were under 30 mg/kg.

Several of the chemical treatments (Tween 20, pH 11/Neodol 91-6, and pH 9/ sellite)

Table 3-11
Summary of Results for Reagent Washing Tests: Gravel Samples

Parameter	Test					
	Tap Water Control	pH 11	pH 11/ Neodol 91-6	pH 9/ Sellite	Tween 20	Macol TD 610
TNT Concentration (mg/kg)						
Cycle 7A	120	17	13	21	4.9	9.4
Cycle 7A Duplicate	14	36	13	14	9.6	44
Cycle 7A Average	67	27	13	18	7.3	27
Cycle 7B	26	28	25	14	17	31
Cycle 7B Duplicate	7	13	3.7 J	7.5	11	7.6
Cycle 7B Average	17	21	14	11	14	19
RDX Concentration (mg/kg)						
Cycle 7A	7.0	0.98	0.28	1.4	1.8	3.2
Cycle 7A Duplicate	0.75	2.5	0.81	3.3	2.3	2.2
Cycle 7A Average	3.9	1.7	0.55	2.4	2.1	2.7
Cycle 7B	13	2.7	0.60	1.1	4.6	1.0
Cycle 7B Duplicate	1.5	0.88	0.60	0.47	3.2	0.84
Cycle 7B Average	7.3	1.8	0.60	0.79	3.9	0.92
HMX Concentration (mg/kg)						
Cycle 7A	2.8	1.0	0.84	0.66	1.4	0.75
Cycle 7A Duplicate	0.59	0.64	0.24 J	1.9	0.83	1.5
Cycle 7A Average	1.7	0.82	0.54	1.3	1.1	1.1
Cycle 7B	2.0	2.2	0.22 J	0.74	2.6	0.62
Cycle 7B Duplicate	0.14 J	1.5	0.10 J	0.75	1.8	0.49
Cycle 7B Average	1.1	1.9	0.16	0.75	2.2	0.56
TS (%)						
Cycle 7A	99.3	99.7	98.2	100	99.8	99.2
Cycle 7A Duplicate	98.8	99.2	97.8	98.6	99.5	98.5
Cycle 7A Average	99.1	99.5	98.0	99.3	99.7	98.9
Cycle 7B	98.5	98.5	98.6	99.7	97.6	99.6
Cycle 7B Duplicate	98.8	99.2	99.2	99.7	98.8	98.6
Cycle 7B Average	98.7	98.9	98.9	99.7	98.2	99.1

"J" indicates compound present below detection limit.

resulted in Cycle 7A and 7B TNT levels that were slightly lower than those of gravel samples treated with pH 11 or Macol TD 610 treatments.

Figure 3-3 illustrates the TNT levels exhibited by gravel samples that were chemically washed using each of the test reagents and with tap water. TNT gravel concentrations after one or two wash cycles (Cycles 7A and 7B, respectively) are shown. For comparison, the TNT concentration of the gravel after the six sequential water washing cycles is also presented. This value represents the concentration of TNT in the gravel prior to chemical washing.

Table 3-11 also shows that for the control sample as well as for each of the chemically treated samples, average RDX levels of gravel samples collected after Cycles 7A and 7B were all well below the 30 mg/kg RDX limit. Average RDX levels of gravel samples collected during the control (tap water) test after Cycles 7A and 7B were 3.9 and 7.3 mg/kg, respectively. Average RDX levels in gravel samples collected after Cycles 7A and 7B during tests in which chemical reagents were used ranged between 0.55 and 3.9 mg/kg. The pH 11/Neodol 91-6 treatment resulted in the lowest average gravel RDX levels (0.55 and 0.60 mg/kg RDX after Cycles 7A and 7B, respectively), although there appeared to be little significant difference in the values produced by treatment with the other chemical reagents.

Figure 3-4 illustrates the RDX levels exhibited by gravel samples that were chemically washed using test reagents and tap water. RDX gravel concentrations after one and two cycles are shown. The figure also contains the RDX gravel concentration prior to chemical washing.

According to Table 3-11, HMX levels of gravel samples collected after Cycles 7A and 7B from tests used to evaluate the control as well as each of the chemical reagents ranged from 0.16 to 2.2 mg/kg. Again, gravel samples treated with the pH 11/Neodol 91-6 combination exhibited the lowest average HMX levels after Cycles 7A and 7B (0.54 and 0.16 mg/kg HMX, respectively), although no significant differences between the HMX values produced by treatment with the control or the other chemical reagents were evident. No cleanup criteria for HMX are established.

Figure 3-3
TNT Concentration in Gravel Samples following Reagent Washing

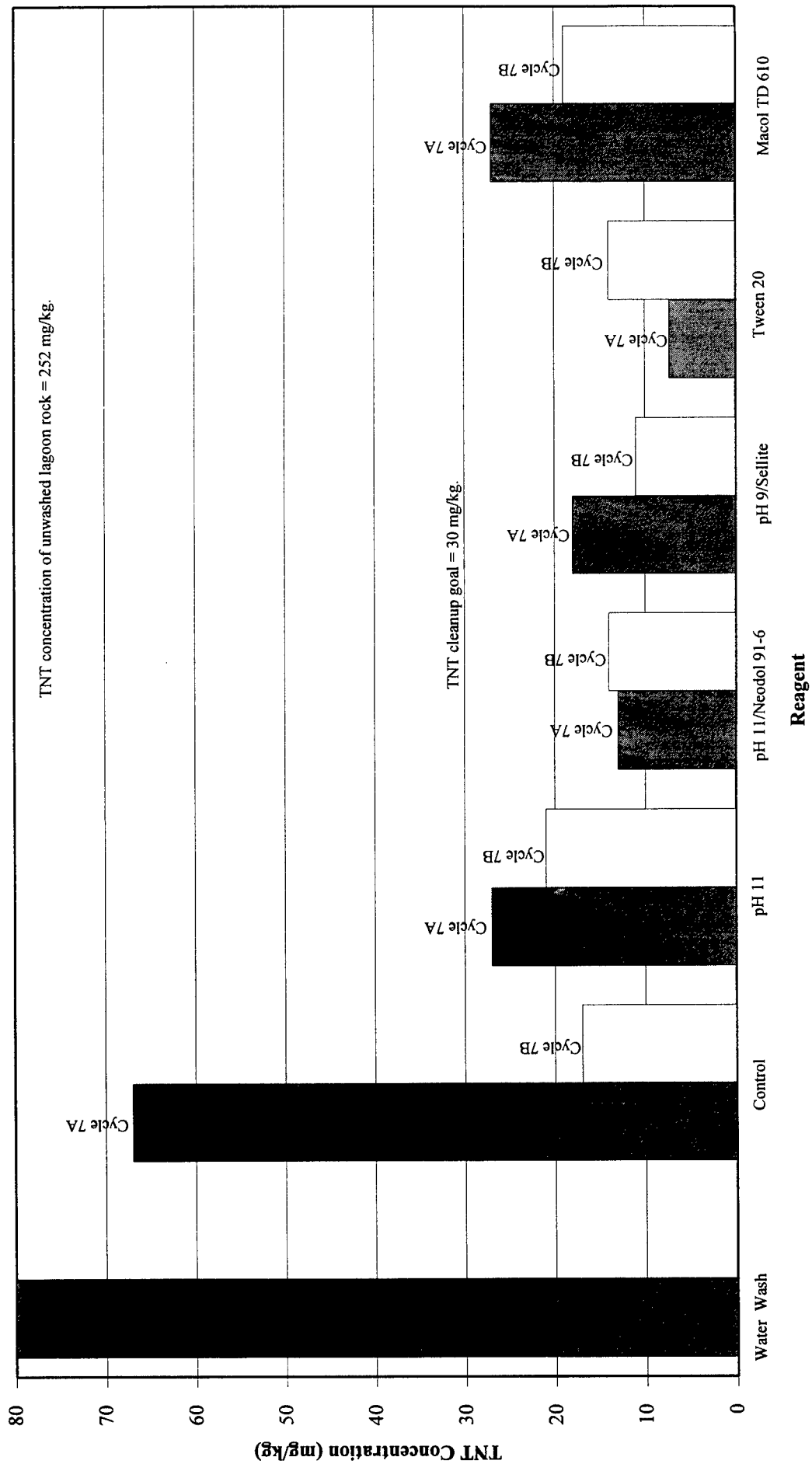


Figure 3-4
RDX Concentration in Gravel Samples following Reagent Washing

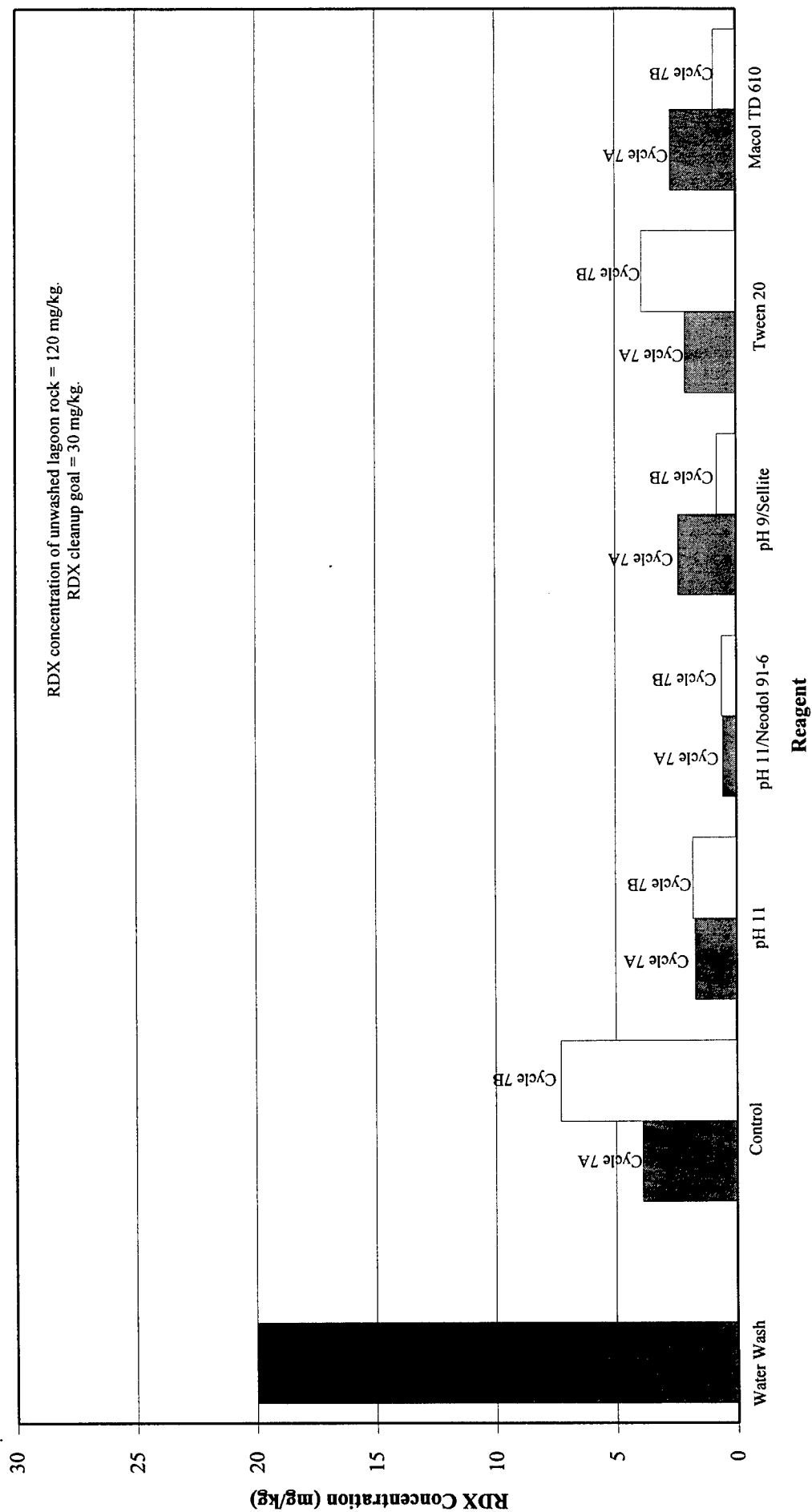


Figure 3-5 illustrates the HMX levels of gravel samples that were chemically washed. HMX gravel concentrations after one and two chemical wash cycles are shown. Finally, Table 3-11 indicates that the total solids levels of all of the samples at the time of testing ranged from 98 to 100%.

Table 3-12 presents a summary of chemical characterization results of wash and rinse liquid samples from the chemical treatment tests. Within each cycle, the levels of explosives present in the wash samples generally were greater than those of the corresponding rinse samples. Cycle 7A wash liquid TNT levels ranged from 13 mg/L to 160 mg/L, while rinse liquid TNT levels ranged from 5.2 mg/L to 42 mg/L. Cycle 7A wash liquid RDX levels ranged from 4.6 to 7.9 mg/L, and rinse liquid RDX samples ranged from 1.5 to 16 mg/L. Cycle 7A HMX levels ranged from 2.6 to 5.1 mg/L, and rinse liquid samples ranged from 1.4 to 15 mg/L. Cycle 7B wash liquid TNT levels ranged from 5.1 to 9.9 mg/L, while rinse liquid TNT levels ranged from 3.4 to 4.5 mg/L. Cycle 7B wash liquid RDX levels ranged from 1.7 to 4.9 mg/L, while rinse liquid RDX levels ranged from 0.90 to 1.5 mg/L. Cycle 7B wash liquid HMX levels ranged from 1.3 to 1.9 mg/L, while rinse liquid levels ranged from 0.89 to 1.2 mg/L. Thus, it can be seen that for each of the six chemical wash tests, the Cycle 7B wash and rinse liquid concentrations of TNT, RDX, and HMX were lower than the corresponding Cycle 7A values. This indicates that increasing the number of wash and rinse steps (regardless of chemical treatment) would likely remove smaller amounts of additional explosive quantities. Additionally, Cycle 7A wash TSS values ranged from 5,570 to 11,000 mg/L while cycle 7A rinse TSS values ranged from 3,840 to 8,260 mg/L. Cycle 7B wash TSS values ranged from 4,690 to 7,640 mg/L with rinse values of 3,720 to 24,800 mg/L.

Table 3-13a contains a material balance for the first wash cycle (Cycle 7A) of each of the six chemical treatment evaluation tests. The table lists both liquid (wash and rinse liquid) and solid (water washed gravel) input quantities for each test. Output quantities for each stream (wash/rinse liquids, gravel, and fines) are also listed separately for each test. Recoveries are presented for liquid and solids are shown. The table also contains overall recovery values.

Figure 3-5
HMX Concentration in Gravel Samples following Reagent Washing

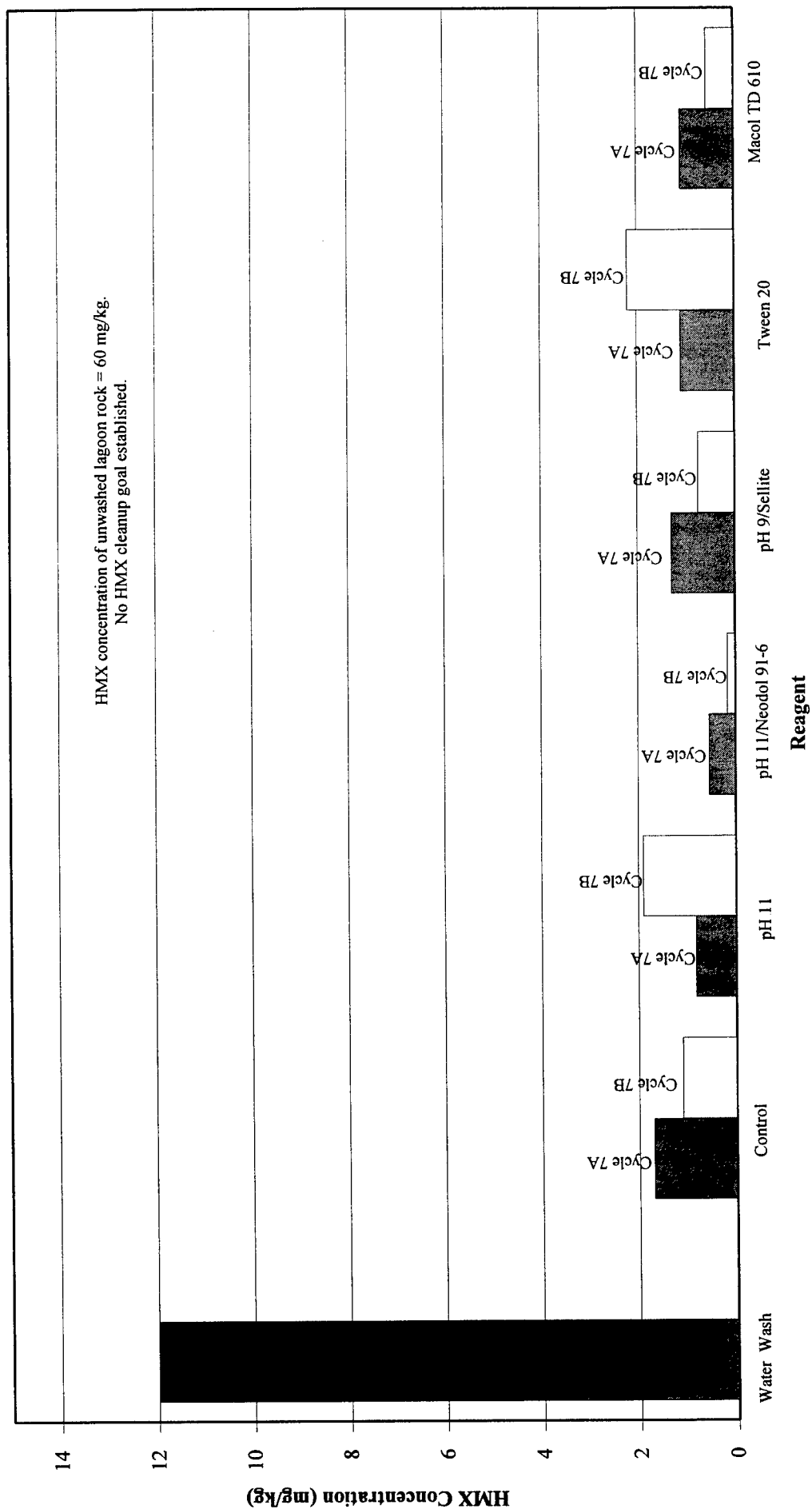


Table 3-12
Summary of Results for Reagent Washing Tests: Liquid Samples

Cycle	Stage	Test											
		Control (Tap Water)						pH 11					
		TNT (mg/L)	RDX (mg/L)	HMX (mg/L)	TSS (mg/L)	TNT (mg/L)	RDX (mg/L)	HMX (mg/L)	TSS (mg/L)	TNT (mg/L)	RDX (mg/L)	HMX (mg/L)	TSS (mg/L)
7A	Wash	18	6.4	3.2	6,260	13	5.4	2.7	9,730	160	7.9	5.1	5,570
	Rinse	8.5	3.7	2.2	8,260	7.3	2.6	2.2	6,200	5.2	1.5	1.4	5,210
7B	Wash	9.9	4.9	1.9	5,210	6.6	2.0	1.3	7,640	5.4	1.7	1.9	5,590
	Rinse	4.4	1.5	1.1	8,550	4.0	1.2	1.1	5,020	4.5	1.1	1.2	4,440

Cycle	Stage	Test											
		pH 9/Sellite						Tween 20					
		TNT (mg/L)	RDX (mg/L)	HMX (mg/L)	TSS (mg/L)	TNT (mg/L)	RDX (mg/L)	HMX (mg/L)	TSS (mg/L)	TNT (mg/L)	RDX (mg/L)	HMX (mg/L)	TSS (mg/L)
7A	Wash	130	4.6	2.6	11,000	15	6.9	3.2	5,970	14	6.3	3.7	10,900
	Rinse	6.6	2.2	1.9	3,840	42	1.6	1.5	5,210	6.1	2.0	1.7	6,200
7B	Wash	8.2	2.1	1.5	4,690	5.1	1.8	1.5	6,240	6.8	2.1	1.9	5,800
	Rinse	4.2	1.2	1.0	3,720	3.4	0.97	1.0	10,900	3.8	0.90	0.89	24,800

Table 3-13a
Material Balance for Reagent Washing Tests: Cycle 7A

Parameter	Test					
	Tap Water Control	pH 11	pH 11/ Neodol 91-6	pH 9/ Sellite	Tween 20	Macol TD 610
Input (kg)						
Liquid	4.484	4.628	4.684	4.528	4.688	4.512
Gravel	8.408	8.676	8.784	8.488	8.786	8.458
Total	12.892	13.304	13.468	13.016	13.474	12.970
Output (kg)						
Liquid	4.330	4.492	3.316	4.302	4.584	3.227
Gravel	8.344	8.596	8.748	8.452	8.750	8.432
Fines	0.0318	0.0358	0.0177	0.0321	0.0257	0.0247
Total	12.706	13.124	12.082	12.786	13.360	11.684
Recovery (%)						
Liquid	96.6	97.1	70.8	95.0	97.8	71.5
Solids	99.6	99.5	99.8	100.0	99.9	100.0

Table 3-13b
Material Balance for Reagent Washing Tests: Cycle 7B

Parameter	Test					
	Tap Water Control	pH 11	pH 11/ Neodol 91-6	pH 9/ Sellite	Tween 20	Macol TD 610
Input (kg)						
Liquid	4.201	4.320	4.480	4.336	4.508	4.336
Gravel	7.880	8.100	8.396	8.128	8.452	8.132
Total	12.081	12.420	12.876	12.464	12.960	12.468
Output (kg)						
Liquid	4.012	4.181	2.964	4.164	4.374	3.073
Gravel	7.854	8.046	8.378	8.080	8.426	8.086
Fines	0.0279	0.0265	0.0144	0.0175	0.0380	0.0590
Total	11.894	12.254	11.356	12.262	12.838	11.218
Recovery (%)						
Liquid	95.5	96.8	66.2	96.0	97.0	70.9
Solids	100.0	99.7	100.0	99.6	100.1	100.2

Table 3-13a shows that, with the exception of the tests used to evaluate the pH 11/Neodol 91-6 and Macol TD 610 treatments, all the calculated liquid recoveries ranged from 95.0% to 97.8%. The liquid recoveries for the pH 11/Neodol 91-6 and Macol TD 610 treatment evaluation tests were 70.8% and 71.5% respectively. These relatively low liquid recovery values are due to the fact that significant quantities of wash liquid were lost during each of these tests due to excessive foam generation. No significant quantities of foam were generated in any of the other wash tests. Solids recoveries ranged from 99.6% to 100.0% for each of the six chemical wash tests.

Table 3-13b summarizes material balances for the second wash cycle (Cycle 7B) of the chemical treatment evaluation tests. The results for Cycle 7B are almost identical to those of Cycle 7A. It can be seen that the liquid recoveries for the pH 11/Neodol 91-6 and Macol TD 610 treatment evaluation tests were 66.2% and 70.9%, respectively, while those for the rest of the chemical wash tests ranged from 95.5% to 97.0%. Solids recoveries ranged from 99.6% to 100.2%.

Tables 3-14a and 3-14b contain mass balance and calculated removals for TNT in Cycles 7A and 7B respectively, of each of the chemical washing agent evaluation tests. Input quantities in these tables include liquid (wash liquid, i.e. tap water both alone and in solution with chemical reagents) and solids (water washed gravel in the case of Cycle 7A, and chemically washed gravel in the case of Cycle 7B). Output quantities include wash and rinse liquid, and gravel. Table 3-14a shows that a recovery of 98.4% was achieved for Cycle 7A of the tap water control test. In contrast, Cycle 7A chemical reagent wash test recoveries ranged from 33.7% to 72.6%. Overall % TNT removal was lowest in Cycle 7A for the tap water control (17.7% TNT removal), and ranged from 67.1 to 91% removal for the chemical treatment tests. The Tween 20-treated samples exhibited the greatest TNT removal in Cycle 7A. According to Table 3-14b, Cycle 7B TNT varied widely and ranged from 38.5% (tap water control) to 319.9% (Tween 20). Overall TNT removals ranged from -89.7% (Tween 20) to 75.6% (tap water control).

Table 3-14a
TNT Mass Balance for Reagent Washing Tests: Cycle 7A

Parameter	Test					
	Tap Water Control	pH 11	pH 11/ Neodol 91-6	pH 9/ Sellite	Tween 20	Macol TD 610
Input (mg)						
Liquid	0	0	0	0	0	0
Gravel	672.6	694.1	702.7	679.0	702.9	676.6
Total	672.6	694.1	702.7	679.0	702.9	676.6
Output (mg)						
Wash Liquid	59.4	62.8	174.7	318.2	55.9	30.7
Rinse Liquid	48.6	40.1	31.1	27.8	117.5	36.5
Gravel	553.7	226.5	111.4	146.9	63.2	222.5
Total	661.8	329.5	317.2	492.9	236.6	289.7
Recovery (%)						
Total	98.4	47.5	45.1	72.6	33.7	42.8
Removal (%)						
Total	17.7	67.4	84.1	78.4	91.0	67.1

Table 3-14b
TNT Mass Balance for Reagent Washing Tests: Cycle 7B

Parameter	Test					
	Tap Water Control	pH 11	pH 11/ Neodol 91-6	pH 9/ Sellite	Tween 20	Macol TD 610
Input (mg)						
Liquid	0	0	0	0	0	0
Gravel	522.9	213.5	107.0	141.2	61.1	214.6
Total	522.9	213.5	107.0	141.2	61.1	214.6
Output (mg)						
Wash Liquid	36.0	38.7	14.6	32.1	32.6	15.8
Rinse Liquid	37.3	25.9	22.7	21.7	46.9	93.4
Gravel	127.8	163.0	118.9	86.6	115.8	154.7
Total	201.2	227.6	156.3	140.4	195.3	263.9
Recovery (%)						
Total	38.5	106.6	146.1	99.4	319.9	122.9
Removal (%)						
Total	75.6	23.6	-11.2	38.7	-89.7	27.9

Tables 3-15a and 3-15b contain RDX mass balances and calculated removals for Cycles 7A and 7B of the chemical washing tests. According to Table 3-15a, overall RDX recovery for Cycle 7A ranged from 51.2% (pH 11/Neodol 91-6) to 110.5% (Tween 20). The tap water control exhibited a recovery of 32.3%. Overall RDX removal for Cycle 7A ranged from 80.8% (tap water control) to 97.3% (pH 11/Neodol 91-6).

Table 3-15b shows that the overall RDX recovery for Cycle 7B ranged from 453.3% (pH 9/sellite) to 1,529.8% (pH 11/Neodol 91-6). Overall RDX removal for Cycle 7B ranged from -86.9% (Tween 20) to 66.7% (pH 9/ sellite). Again, the discrepancies in Cycle 7B are probably due to a lack of fines explosives data.

Tables 3-16a and 3-16b contain mass balance and calculated removals for HMX in Cycles 7A and 7B, respectively, of the chemical wash tests. According to Table 3-16a, overall recovery for Cycle 7A ranged from 45.3% (pH 11/Neodol 91-6) to 97.0% (Tween 20). Overall HMX removal for Cycle 7A ranged from 86.1% (tap water control) to 95.6% (pH 11/Neodol 91-6).

Table 3-16b shows that the overall HMX recovery for Cycle 7B ranged from 439.5 (pH 9/sellite) to 1,081.7% (pH 11). Overall HMX removal for Cycle 7B ranged from -122.8% (pH 11) to 70.2% (pH 11/Neodol 91-6).

As shown in Table 3-13, closure on the mass balance for total solids was generally excellent with total solids recoveries ranging from 99.5% to 100.2%. However, as shown in Tables 3-14a through 3-16b, closure on mass balances for the individual explosives are less satisfactory. Since the high TSS levels in the washwater were not anticipated, explosives analyses were not conducted on these solids fractions. Consequently, for purposes of these mass balance calculations, the limited explosives analyses for the fines fraction from the initial water washing steps (average of 2 analyses) were used as the assumed explosives concentrations for all mass balances. This may introduce some error in the mass balance calculation to the extent that the final washwater fines may not exhibit explosives levels similar to the original fines. It is possible that after contact with surfactants, the final

Table 3-15a
RDX Mass Balance for Reagent Washing Tests: Cycle 7A

	Test					
	Tap Water Control	pH 11	pH 11/ Neodol 91-6	pH 9/ Sellite	Tween 20	Macol TD 610
Input (mg)						
Liquid	0	0	0	0	0	0
Gravel	168.2	173.5	175.7	169.8	175.7	169.2
Total	168.2	173.5	175.7	169.8	175.7	169.2
Output (mg)						
Wash Liquid	68.7	100.6	32.1	108.3	72.1	50.3
Rinse Liquid	85.1	66.0	53.2	39.5	87.8	63.1
Gravel	32.0	14.9	4.7	19.7	17.9	22.5
Total	185.8	181.5	89.9	167.6	177.8	135.9
Recovery (%)						
Total	110.5	104.6	51.2	98.7	101.2	80.3
Removal (%)						
Total	81.0	91.4	97.3	88.4	89.8	86.7

Table 3-15b
RDX Mass Balance for Reagent Washing Tests: Cycle 7B

Parameter	Test					
	Tap Water Control	pH 11	pH 11/ Neodol 91-6	pH 9/ Sellite	Tween 20	Macol TD 610
Input (mg)						
Liquid	0	0	0	0	0	0
Gravel	30.2	14.0	4.5	19.0	17.3	21.7
Total	30.2	14.0	4.5	19.0	17.3	21.7
Output (mg)						
Wash Liquid	52.4	69.3	25.4	44.1	59.9	25.8
Rinse Liquid	76.3	47.4	38.2	35.6	104.0	223.0
Gravel	56.2	14.2	5.0	6.3	32.3	7.4
Total	184.9	131.0	68.6	86.0	196.1	256.1
Recovery (%)						
Total	611.4	934.3	1529.8	453.3	1135.8	1180.1
Removal (%)						
Total	-85.7	-1.6	-10.9	66.7	-86.9	66.0

Table 3-16a
HMX Mass Balance for Reagent Washing Tests: Cycle 7A

Parameter	Test					
	Tap Water Control	pH 11	pH 11/ Neodol 91-6	pH 9/ Sellite	Tween 20	Macol TD 610
Input (mg)						
Liquid	0	0	0	0	0	0
Gravel	100.9	104.1	105.4	101.9	105.4	101.5
Total	100.9	104.1	105.4	101.9	105.4	101.5
Output (mg)						
Wash Liquid	32.7	47.6	16.5	46.2	33.8	24.4
Rinse Liquid	41.0	33.3	26.6	20.4	58.7	31.4
Gravel	14.0	7.0	4.6	10.7	9.7	9.4
Total	87.7	87.9	47.7	77.4	102.2	65.1
Recovery (%)						
Total	86.9	84.5	45.3	76.0	97.0	64.2
Removal (%)						
Total	86.1	93.3	95.6	89.5	90.8	90.8

Table 3-16b
HMX Mass Balance for Reagent Washing Tests: Cycle 7B

Parameter	Test					
	Tap Water Control	pH 11	pH 11/ Neodol 91-6	pH 9/ Sellite	Tween 20	Macol TD 610
Input (mg)						
Liquid	0	0	0	0	0	0
Gravel	13.2	6.6	4.4	10.3	9.4	9.0
Total	13.2	6.6	4.4	10.3	9.4	9.0
Output (mg)						
Wash Liquid	23.8	33.3	13.1	21.8	29.6	13.0
Rinse Liquid	36.7	23.4	19.3	17.6	50.1	105.8
Gravel	8.3	14.7	1.3	6.0	18.2	4.4
Total	68.8	71.5	33.7	45.4	97.8	123.2
Recovery (%)						
Total	520.1	1081.7	758.1	439.5	1041.7	1362.7
Removal (%)						
Total	37.3	-122.8	70.2	41.9	-93.8	50.8

washwater fines may exhibit lower explosives levels than those used in these mass balance calculations. The variations associated with the TSS measurements in the wash and rinse liquids also may have contributed to the lack of closure for the explosives mass balances.

3.3 CONCLUSIONS

The following conclusions summarize the results and key findings of the Phase II bench-scale reagent washing treatability study for rocks from explosives washout lagoons at UMDA:

Untreated Rock

- The as-received, unwashed lagoon rock samples consisted of gray, poorly graded gravel containing approximately 3% to 5% sand and 0% to 1% silt. The USCS designation of the material was GP. The as-received, unwashed lagoon rock samples had a solids content of approximately 99.5% and a wet and dry bulk density of approximately 105 lb/ft³.
- The average TNT, RDX, and HMX concentrations in the as-received, unwashed lagoon rock were 252 mg/kg, 120 mg/kg, and 60 mg/kg, respectively. The relative proportions of explosives were 58% TNT, 28% RDX, and 14% HMX. There were significant differences in the amount of explosives contamination of the rock batches received for use in Phase I and II of the rock washing study. The rocks used in Phase I exhibited much higher levels of contamination than those used in Phase II.

Water-Based Washing

- After six, 30-minute water washing cycles, average gravel concentrations of TNT, RDX, and HMX were all decreased compared to those of unwashed rocks. The average TNT, RDX, and HMX concentrations in the washed gravel fraction after six 30-minute water washing cycles were 80 mg/kg, 20 mg/kg, and 12 mg/kg respectively. The UMDA cleanup criteria for both TNT and RDX is 30 mg/kg. As a result, the TNT cleanup goal for the water washed rocks was not met.
- Explosives levels increased as the particle size range of the water washed solids fraction decreased. Thus, explosives concentrations were highest in the washed fines fraction, intermediate in the washed sand fraction, and lowest in the washed gravel. The average TNT, RDX, and HMX concentrations in

the washed sand fraction after six 30-minute water washing cycles were 303 mg/kg, 163 mg/kg, and 26 mg/kg, respectively. The average TNT, RDX, and HMX concentrations in the washed fines fraction after six, 30-minute water washing cycles were 1,168 mg/kg, 2,821 mg/kg, and 1,309 mg/kg, respectively.

- The average TSS concentration in the composited wash water after six 30-minute water washing cycles was 9,300 mg/L.
- The TNT, RDX, and HMX concentrations in the composited wash water filtrate after six 30-minute water washing cycles were 43 mg/L, 25 mg/L, and 6.6 mg/L, respectively.

Qualitative Screening Tests

- Webster's reagent was an effective indicator for the presence of TNT and gave positive results when used to test rocks washed using the control treatment (tap water) as well as each of the chemical treatments evaluated in the study. Therefore, no wash system was completely effective in removing residual TNT from rock surfaces. Because the sensitivity of Webster's reagent in the detection of TNT on solids is not definitely known, it is not known whether residual TNT levels are below the 30 ppm treatment criterion. Webster's reagent was also effective in detecting the presence of explosives in aqueous (washate and rinsate) samples. Observation of these samples, both with and without Webster's reagent added, were used to determine the relative rankings of chemical treatments.
- Diphenylamine/sulfuric acid was not an effective indicator for the presence of RDX in this application since the characteristic blue color did not develop in any case tested including contaminated, unwashed rocks.
- Based on the results of the qualitative screening study, and on correspondence and conversations with WESTON and USAEC personnel, the following chemical treatments were selected for inclusion in the final reagent test evaluations:
 - a) Tap water control
 - b) pH 11
 - c) pH 11/Neodol 91-6
 - d) pH 9/sellite
 - e) Tween 20
 - f) Macol TD 610

Reagent Washing

- The average TNT level of gravel samples collected from the tap water control test was 67 mg/kg after one chemical wash cycle (Cycle 7A) and 17 mg/kg after two chemical wash cycles (Cycle 7B). This indicates that up to eight 30-minute water washes were ineffective in reducing washed gravel TNT levels below the TNT cleanup criterion of 30 mg/kg. However, ten 30-minute water washes were effective in achieving the 30 mg/kg TNT cleanup goal.
- Gravel samples treated with each of the chemical washing reagents exhibited average TNT levels ranging from 7.3 mg/kg to 27 mg/kg after the first chemical wash cycle. These values were all below the TNT cleanup goal of 30 mg/kg. After two chemical washes, each of the chemically treated gravel samples exhibited average TNT levels ranging from 11 mg/kg to 21 mg/kg. Overall, each of the chemical wash treatments resulted in a decrease in gravel TNT levels compared to that of the control test after one chemical wash cycle. None of the chemical treatments provided any significant reduction in gravel TNT levels relative to the control treatment after two chemical wash cycles. There appeared to be no significant reduction in gravel TNT levels after two chemical washes compared to gravel TNT levels already achieved after one chemical wash cycle using the same chemical treatment.
- The average RDX level of gravel samples collected from the tap water control test was 3.9 mg/kg after one chemical wash cycle and 7.3 mg/kg after two chemical wash cycles. Thus, the use of two additional 30-minute water washes reduced gravel RDX levels compared to those achieved after the initial six 30-minute water washes. However, additional washing for RDX reduction was not necessary after the first six 30-minute water washing cycles since the RDX levels achieved (20 mg/kg) were already below the RDX cleanup goal of 30 mg/kg.
- The average RDX levels of gravel samples collected from the chemical treatment tests ranged from 0.55 mg/kg to 2.7 mg/kg after one chemical wash cycle, and from 0.60 mg/kg to 3.9 mg/kg after two chemical wash cycles. These values were all below the RDX cleanup goal of 30 mg/kg. Each of the chemical washing treatments resulted in a very slight decrease in gravel RDX levels compared to that of the control test after one chemical wash cycle. Also, each of the chemical washing treatments produced a slight decrease in gravel RDX levels after two chemical wash cycles compared to that of the control test after two chemical wash cycles. There appeared to be no significant reduction in gravel RDX levels after two chemical washes compared to gravel RDX levels already achieved after one chemical wash cycle using the same chemical treatment.
- The average HMX level of gravel samples collected from the tap water control test was 1.7 mg/kg after one chemical wash cycle and 1.1 mg/kg after

two chemical wash cycles. Thus, the use of two additional 30-minute water washes reduced gravel HMX levels compared to the HMX levels achieved after the initial six 30-minute water washes. However, additional washing for HMX reduction may not be necessary since no cleanup goal for HMX has been established. When using any of the chemical reagents, there appeared to be no significant reduction in gravel HMX levels after two chemical washes compared to gravel HMX levels already achieved after one chemical wash cycle using the same chemical treatment.

SECTION 4

HOT GAS DECONTAMINATION EVALUATION

4.1 INTRODUCTION

USAEC has investigated technologies that would effectively treat explosives-contaminated components. Hot Gas Decontamination is one of those technologies. Based on hot gas pilot studies conducted by USAEC at Hawthorne Army Ammunition Plant (HWAAP) in 1989, the hot gas decontamination process was found to provide an effective means of decontaminating explosives-contaminated test items (i.e., clay pipe, shell casings, etc.) prior to disposal or reuse of those items (6).

In the following subsections, the potential viability of a hot gas decontamination system for the treatment of explosives-contaminated rock at sites such as the UMDA washout lagoons will be discussed. The major points of discussion will focus on the items listed below:

- Review of the existing pilot studies to determine treatment temperatures, retention times, and required equipment.
- Determine whether commercially available equipment exists.
- Discuss potential costs associated with this equipment.
- Conclusions of the evaluation.

4.2 BACKGROUND

USAEC conducted pilot studies at the Hawthorne Army Ammunition Plant (HWAAP) between 10 July and 21 September 1989 to evaluate the feasibility of Hot Gas Decontamination on test items contaminated with TNT by determining the destruction and removal efficiency (DRE) of the hot gas process for TNT.

The pilot study equipment was supplied by the government and consisted of an air preheater, flash chamber, and afterburner arranged in the configuration shown in Figure 4-1.

Nine test runs with TNT-contaminated items were conducted. All bulk contamination had been removed from the test items prior to treatment to eliminate possible high-pressure conditions caused by auto-ignition events in the flash chamber.

Results from the pilot tests indicated that a minimum temperature of 500°F and residence time of 6 hours were required to effectively decontaminate the test items contaminated with TNT using the system shown in Figure 4-1. These test results indicated that items treated in this manner are no longer considered hazardous and may safely be reused or disposed. Stack test results indicate the DRE for TNT exceeded 99.99% when measurable levels of TNT were emitted from the afterburner (during same operations, TNT could not be detected in the exhaust).

Based on these results, it is possible that a hot gas decontamination system will provide an effective way to decontaminate TNT and RDX contaminated rocks such as that removed from the UMDA washout lagoons.

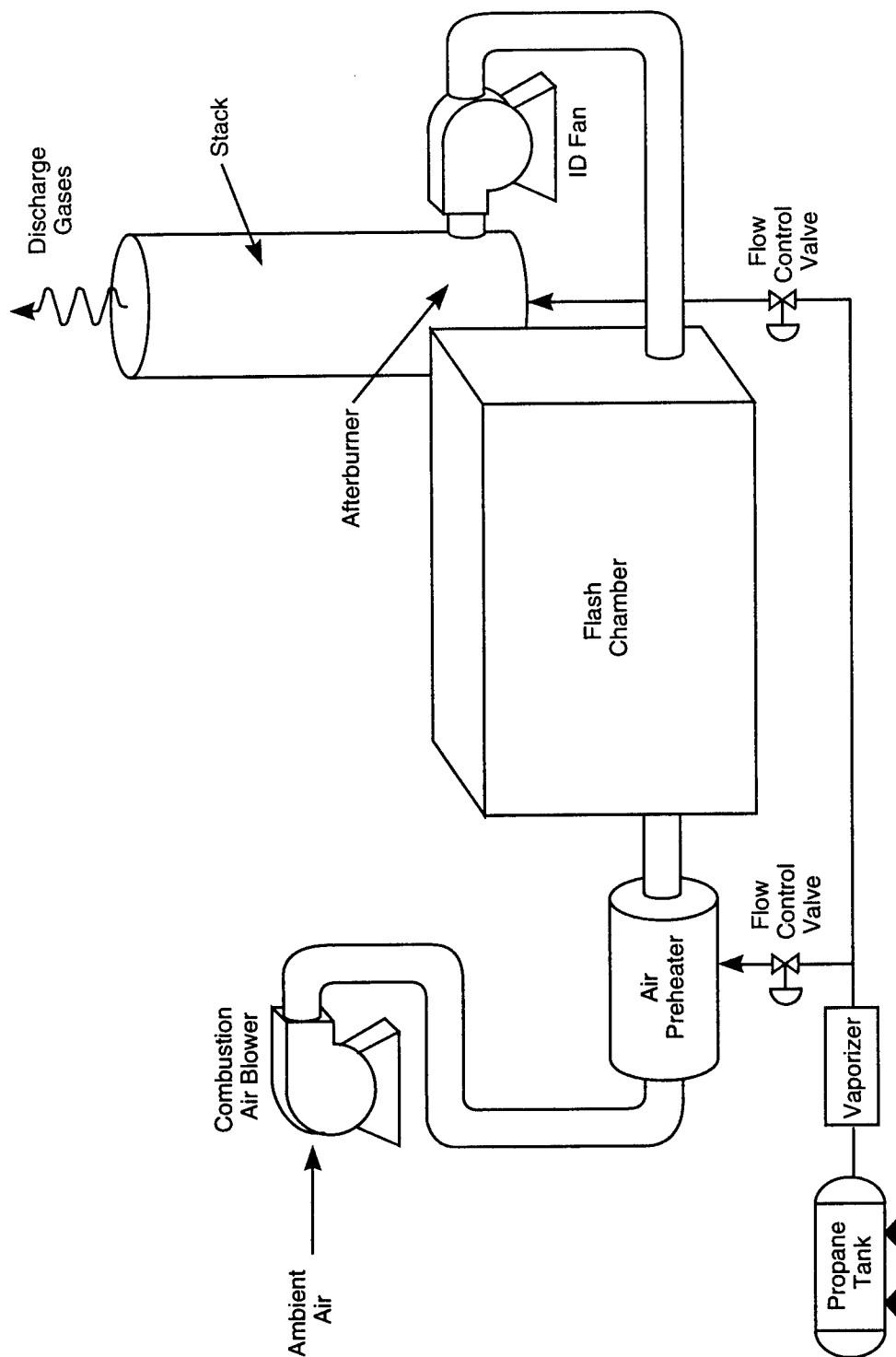
4.3 IDENTIFICATION OF POTENTIAL SYSTEMS

Although the pilot test equipment at HWAAP was effective, pilot study recommendations indicated that modifications to the pilot test equipment could increase the overall process efficiency and possibly reduce the destruction temperature and residence time necessary for effective decontamination. Based on this recommendation two different categories of equipment were considered for this study:

- Equipment schemes similar to the process equipment used in the pilot study.
- Equipment schemes which differ from the pilot study equipment but are capable of meeting the treatment temperature and retention time requirements.

All equipment schemes investigated had to conform to the following minimum requirements:

- Capable of reaching 500°F minimum destruction temperature.



1622-5335 11/16/93

Figure 4-1: Schematic Diagram for Process Equipment at HWAAP

- Capable of maintaining $\geq 500^{\circ}\text{F}$ for 6 hours.
- Gas/propane fired burner system.
- Afterburner for process off-gas treatment.
- Skid mounted and easily transported.
- Minimum instrumentation and controls for safe operations and monitoring.

Lease and/or turnkey options were identified when available. A continuous emissions monitoring (CEM) system for monitoring stack emissions was not included in any of schemes but could easily be incorporated into any of the systems evaluated.

It was assumed the contaminated material to be treated by these equipment systems would fit the general profile identified below:

- Explosives-contaminated rock and gravel ranging in size from approximately $\frac{1}{2}$ " to 2" in diameter.
- Approximately 1,600 tons (3,200,000 lbs) of contaminated gravel and rock to be treated.
- Contaminated material density between 100-120 lb/ft³.
- Contaminated rock and gravel could be rinsed to remove bulk contamination and dirt (although this may not be required).
- Maximum explosives loading of approximately 50 lbs/batch assuming a maximum feed rate of 8,000 lbs of gravel contaminated with approximately 6,000 ppm TNT.

Based on these assumptions and requirements, a telephone survey of thermal treatment and furnace vendors was conducted to identify existing, new, or used equipment which could be used to decontaminate the explosives contaminated gravel and rock at the UMDA lagoons using the hot gas decontamination principles proven at HWAAP. A list of vendors who responded to requests for information is provided in Table 4-1.

Table 4-1
Potential Vendors for Hot Gas Decontamination Systems

Furnace Suppliers:

L&L Special Furnace Company, Inc.
20 Kent Road
P.O. Box 2129
Aston, PA 19014-1494 (215) 459-9126

Keith Company, Inc.
Lock Lomand at Van Norman Road
Pico Rivera, CA 90660-2588 (800) 545-4567

A.R.T. Studio Clay Company
Alpine Kilns and Furnaces
1555-T Louis Avenue
Elk Grove Village, IL 60007 (708) 593-6060

IRV Systems

TerraChem Environmental Services, Inc.
P.O. Box 410302
Charlotte, NC 28241-0302 (704) 377-4718

Afterburner Systems

Sun Equipment and Engineering Company, Inc.
225 W. Second Street
Bethlehem, PA 18015 (215) 868-3339

Keith Company, Inc.
Lock Lomand at Van Norman Road
Pico Rivera, CA 90660-2588 (800) 545-4567

Fluidized Bed Systems

Procedyne Corporation
11 Industrial Drive
New Brunswick, NJ 08901 (908) 249-8347

4.4 RESULTS

The results of the telephone survey identified three different technologies, based on the hot gas decontamination principles identified at HWAAP, which could be used to effectively treat the contaminated rock and gravel at UMDA. They are:

- Gas-Fired Shuttle Car Furnace with Afterburner
- Portable IRV Hydrocarbon Extractor with Afterburner
- Fluidized Bed Heater with Afterburner

Each of these systems is discussed in detail in the following subsections.

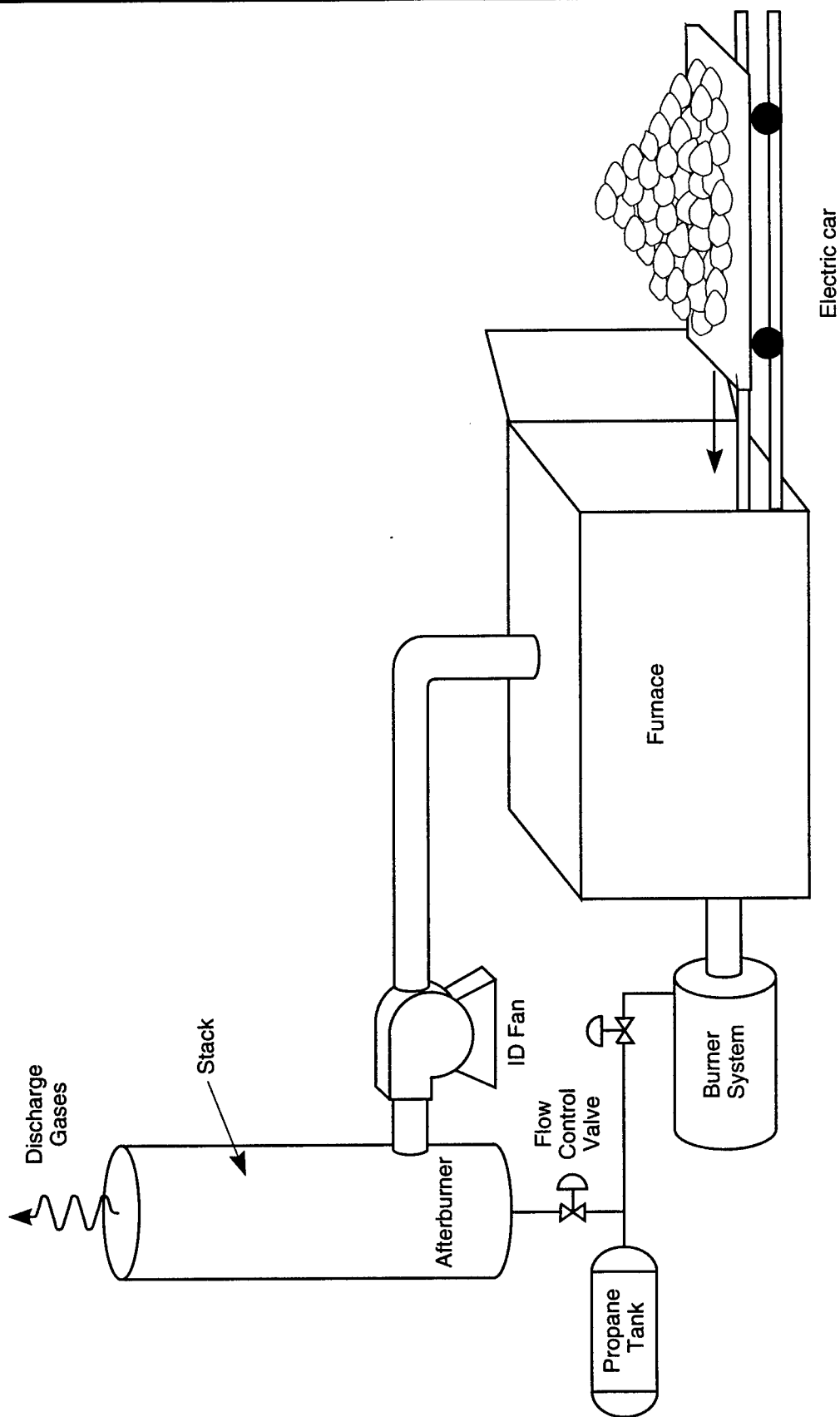
4.4.1 Gas-fired Shuttle Car Furnace

This system is closest to the pilot test equipment used at HWAAP and is illustrated by Figure 4-2. The system equipment includes the following:

- Gas/propane-fired furnace
- Rail-mounted, electrically-operated, furnace car(s)
- Induced draft fan
- Gas/propane-fired afterburner

This system would operate in the same manner as the pilot study equipment used at HWAAP. Contaminated material is loaded onto a furnace car. The car is then moved into the furnace and the furnace is brought up to the 500°F destruction temperature. At the end of the 6-hour hold period, the furnace is cooled and the treated material is removed from the furnace. Process off-gases from the furnace are directed via an induced draft fan to the afterburner which is maintained at 1,800°F throughout the processing time.

The furnace is refractory-lined and is heated by a gas-fired burner system which includes a combustion air blower, gas train, and automatic flame monitor system. Gas and combustion air supplies to the burner are automatically controlled to maintain furnace uniformity at low temperatures. The burner is capable of maintaining 1,000°F maximum with a maximum



1622-5335C 11/10/93

Figure 4-2: Gas-Fired Shuttle Car Furnace Schematic

loading of 6,000 lbs of contaminated rocks and gravel for the required 6 hour hold time. Furnace temperature and hold times are controlled and monitored automatically throughout the process time via a programmable logic controller. Process temperatures and hold times are easily adjusted to suit the required operating parameters. The furnace car is rail-mounted, electrically-controlled, and refractory-lined. The furnace can be supplied with two cars so that one car can be off-loaded and loaded while the other car is in the furnace.

The operating parameters utility requirements for the furnace and afterburner systems would include the following :

- Voltage: 460/3/60
- Natural Gas: 1 psi or more, connected pressure
- Btus: 1,500,000/hour for the burner
900,000/hour for the afterburner
- Utility Water

The budgetary costs (provided by one vendor), including the afterburner and operating costs, for this system are presented below. This estimate includes the furnace cost with one furnace car, the induced draft fan and afterburner. This price does not include permitting, system engineering, treated material chemical analysis, or a CEM system. None of the furnace vendors contacted had used or rental equipment available. The operation of the Gas-Fired Shuttle Car Furnace is based on a 24-hour per day schedule utilizing two, 2-man crews. The crews would process a total of three, 2-ton batches per day. This estimate assumes a 2-hour transition time for loading and unloading batches and 6 hours of treatment for each batch. In addition to the furnace, support facilities including an office trailer, backhoe/loader, and portable decon equipment would be required. The estimate includes costs for operating labor and expenses, utilities, and site equipment.

For this estimate, capital costs for the Gas-Fired Shuttle Car Furnace were assumed to be depreciated over the length of the project due to the presumed difficulty in disposing of

used equipment (in the marketplace) after its use as a hazardous waste treatment system. On the basis of this assumption, the costs of the Gas-Fired Shuttle Car Furnace operations would cost \$550 per ton of treated rock. The cost breakdown for this estimate is approximately 55% labor, 25% expenses, and 30% capital service fees.

If a sufficient market and number of potential applications are available for continued use of the Gas-Fired Shuttle Car Furnace, a 5-year standard equipment depreciation schedule could be employed. The reduction in capital charges would reduce treatment costs from \$550 to \$450 per ton of rock. The cost breakdown is approximately 67% labor, 30% expenses, and 3% capital service fees.

While operation of a single unit can complete operations under the assumed treatment scenario in 7 months, the labor utilization during treatment is not optimal. With one unit in operation, the crew is idle for approximately 18 hours per day, assuming 3 batches per day and 6-hour processing times per batch. Only 6 hours per day is active labor loading and unloading the furnace. The use of multiple units would improve the efficiency of the labor and achieve reduced production time. With two units operating on a staggered schedule, the total operations costs per ton would be reduced from \$550 to \$245 per ton. The cost breakdown for the multiple-unit operations consist of 62% labor, 32% expenses, and 6% capital service fees.

A building or structure would need to be provided to protect the furnace from the weather and is included as an optional addition. Cost for mobilization/demobilization and a simple structure to protect the operating unit from rain would add approximately \$115,000 to the total project cost, or approximately \$72/ton of rock treated.

Using a larger furnace would also increase furnace loading and thereby reduce the estimated time to complete the project. However, a larger furnace is not easily transportable and the estimated system cost (including operating costs) would likely increase.

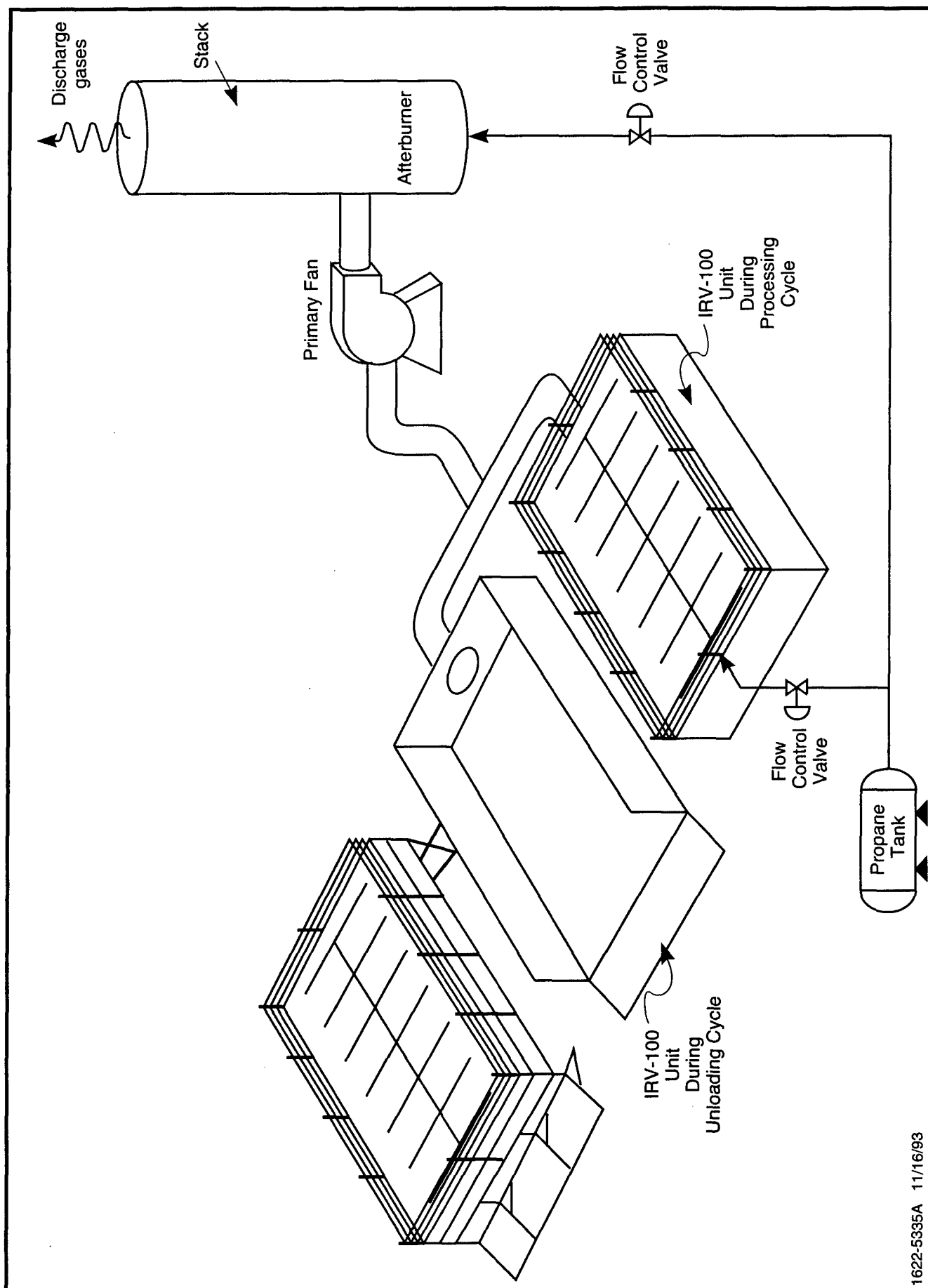
4.4.2 Portable IRV Hydrocarbon Extractor

The IRV-100 hydrocarbon extractor unit is illustrated in Figure 4-3. It is a portable system consisting of the following equipment:

- Vacuum extraction chamber and processor bed,
- Heat cover (which contains the system heaters),
- 16 propane-fired infrared heaters,
- Induced draft fan, and
- Propane-fired afterburner system.

The IRV system is owned and operated by TerraChem Environmental Services, Inc. of Charlotte, North Carolina. The principles used in this method of treatment include: infrared heat, convection heat, vacuum extraction and reduced pressure volatilization in a batch treatment system. Contaminated rock and gravel is placed in the processor bed using a front-end loader. Contaminated solids can be loaded approximately 12-inches deep in the processor bed. Infrared heaters mounted above the contaminated material provide the primary heat source. Infrared heat, with some convection heat from the propane-fired infrared source, heats the top four inches of the contaminated rock and gravel without heating the air in the gap between the rocks and the heating element. Upon striking the rock and gravel the infrared energy is converted to heat and the top layer of rock becomes hot. Process off-gases are drawn downward, through the solids matrix via the vacuum system and the temperature from the top layer of rock and gravel is used to heat the remaining 8-inches of contaminated solids through conduction and convection. The steel top to the extraction chamber is rolled on and off, and the front of the chamber door is opened to allow for easy loading and unloading of the processor. Processor off-gases are directed via a fan into the afterburner. The afterburner operates at 1,600°F in the current configuration. TerraChem's system also includes stack monitoring equipment with a continuous recording system to assure compliance with all air emission standards during processing.

TerraChem will mobilize, setup and operate the IRV equipment on site. TerraChem will provide stockpiling of treated material. The IRV equipment does not require a building or



1622-5335A 11/16/93

Figure 4-3: 100 IRV Hydrocarbon Extractor System

structure to protect it from the elements, although a contaminated feed storage facility would be desirable. Operating requirements and utilities required to support the IRV system include the following:

- Power: 460/3/60 for IRV processors and afterburner
220/3/60 for the blower
120/1/60 for control circuits
- Natural Gas: 5 psig minimum, connected pressure
- Btus: 3,000,000 Btu/hour for dual IRV units
1,800,000 Btu/hour for the afterburner
- Utility Water

Mobilization costs to the UMDA washout lagoons were estimated by the vender to be \$20,000.00. Assuming a process temperature of 600°F and hold time of 6 hours, budgetary processing costs are \$240.00 per ton or \$384,000.00 for 1,600 tons of contaminated solids. TerraChem estimates the project would take 1 month to complete based on operations 6 days/week, 24 hours/day. The total cost of treatment using the IRV system, including mobilization cost, for these conditions would be \$404,000.00. Thus, the total unit cost under this scenario is approximately \$250 per ton of rock.

4.4.3 Fluidized-Bed Heater

The Fluidized Bed Heater system shown in Figure 4-4 consists of the following equipment:

- Fluidized bed reactor
- Air preheater
- Air fan/blower
- Gas-fired afterburner system

The fluidized bed is a cylindrical shell with a grid structure on its bottom surface to support a sand bed. Preheated (fluidizing) air is introduced into the sand bed through the grid at the bottom of the sand bed. The fluidizing air generates a high degree of turbulence into the sand bed and causes the sand to become fluidized. The fluidized air is preheated by

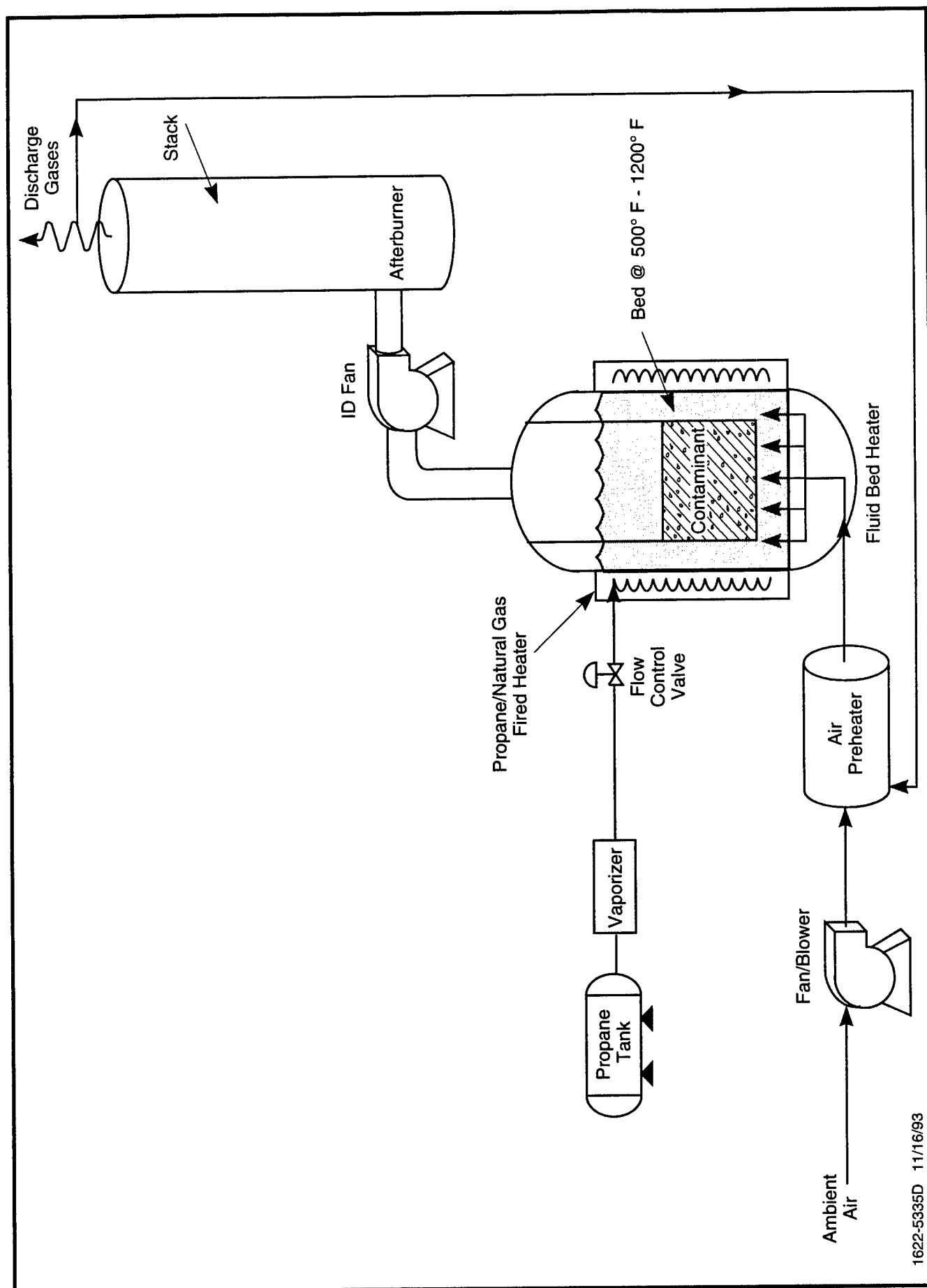


Figure 4-4 Fluidized Bed Heater Schematic

1622-5335D 11/16/93

means of an air preheater. The shell of the fluidized bed is heated via a gas or propane fired burner system. The sand bed can be maintained at temperatures in excess of 1,000°F although the system investigated for this study would normally operate at 850°F to 1,000°F. An induced draft fan is provided to draw process off-gases through the afterburner. Heat generated by the afterburner can be used to preheat the fluidizing air. Contaminated gravel and rock from the washout lagoons would be loaded into a basket and placed into the fluidized bed for processing. Multiple baskets would allow for basket loading and unloading while another is processing.

The fluidized bed equipment would have to be purchased new, leased or used equipment is rare. A building or structure would be required to protect the fluidized bed processor from the elements. The fluidized bed manufacturers contacted would not provide cost information without bench-scale tests because the manufacturers felt the data would not be meaningful. However, all manufacturers thought a fluidized bed system could easily handle the job of volatilizing TNT and RDX contamination found on the rock and gravel from the UMDA washout lagoons. The utilities requirements for this system would be qualitatively similar to the other systems in that electric, natural gas and water must be provided to the site.

The fluidized bed system can be expected to be more efficient than the hot gas pilot test system because the heat transfer coefficient is significantly higher with a fluidized bed system than with other systems. The manufacturers contacted suggested the processing time could be significantly reduced and the need for an afterburner possibly eliminated if bench-scale tests of the contaminated gravel could be conducted. The bench-scale tests would be conducted at the suppliers facility.

Batch sizes for the fluidized bed systems are generally smaller (approximately 1,000 lbs per batch) than with other systems, however, recovery times after loading a new batch are greatly reduced because the sand within the bed acts as a heat sink and the fluidized bed heater is a relatively tight system. The equipment cost for this system is expected to be more expensive than the furnace system. However, fluidized beds in general are known to be extremely efficient because the heat transfer coefficient is significantly reduced, and the

need for an afterburner could possibly be eliminated if bench-scale tests on the contaminated gravel were conducted. The bench-scale tests would be conducted at the suppliers facility. Reduced processing costs expected with the fluidized bed system may partially offset the anticipated high equipment cost associated with this system.

4.5 CONCLUSIONS

The following conclusions are provided based on the evaluation of Hot Gas Decontamination technologies conducted:

- All the systems identified can be used to treat the explosives-contaminated rock and gravel at the UMDA washout lagoons.
- Further testing would be required to verify the optimum operating conditions of the system selected because:
 - The rock and gravel to be treated at UMDA are different from those items decontaminated in the pilot studies at HWAAP.
 - All three of the systems suggested are likely to be more efficient than the pilot study equipment used at HWAAP.
- The system most like the pilot test equipment is the furnace/afterburner system.
- Both the furnace and fluidized bed systems require design, engineering, and purchasing support to procure the necessary equipment. A shelter to house the furnace or fluidized bed should be provided to protect the processor equipment from the weather. A shakedown period would be necessary to verify proper operation of the system, and an operations crew would have to be hired and trained to operate each system. The purchased equipment would be the property of the purchaser, however, both systems are easily transportable and capable of reaching 1000°F which increases the possibility of reusing the equipment at different sites for different contaminants which might require higher volatilization temperatures.
- Assuming the average material size is approximately 2-inches in diameter and the contaminated material to be treated has been excavated from sandy-gravelly soils, it is unlikely the contaminated rock and gravel at UMDA would have to be pre-treated (i.e., rinsed before placement into any of the suggested systems). However, contaminated rock and gravel excavated from more sticky or clayey type soils may require some feed preparation (i.e., screening,

drying, crushing) before treatment in any of the suggested systems. Bench-scale or pilot-scale testing could verify the need for feed preparation.

- A CEM system is provided as part of the IRV system. A CEM system would have to be rented or purchased if stack monitoring is required with the furnace or fluidized bed systems.
- In terms of equipment costs, the fluidized bed processor is likely to be the most expensive option proposed. However, reduced batch processing time and the possibility of eliminating the afterburner based on bench-scale testing by the vendor may make the expected high initial equipment cost more attractive over the life of the project.

SECTION 5

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